Chemical Exposition Daily, Sept. 25

CHEMICAL & METALLURGICAL ENGINEERING

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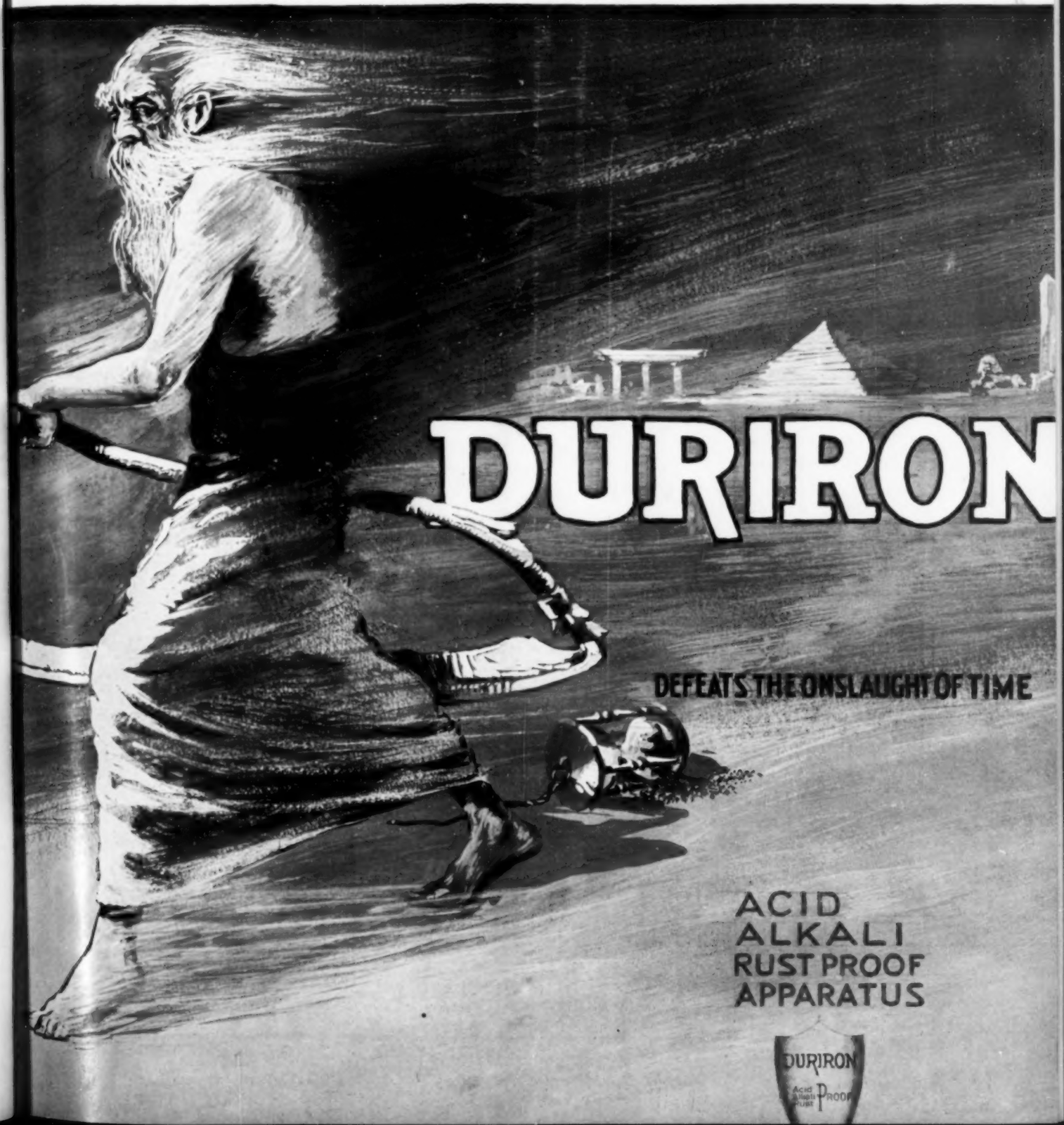
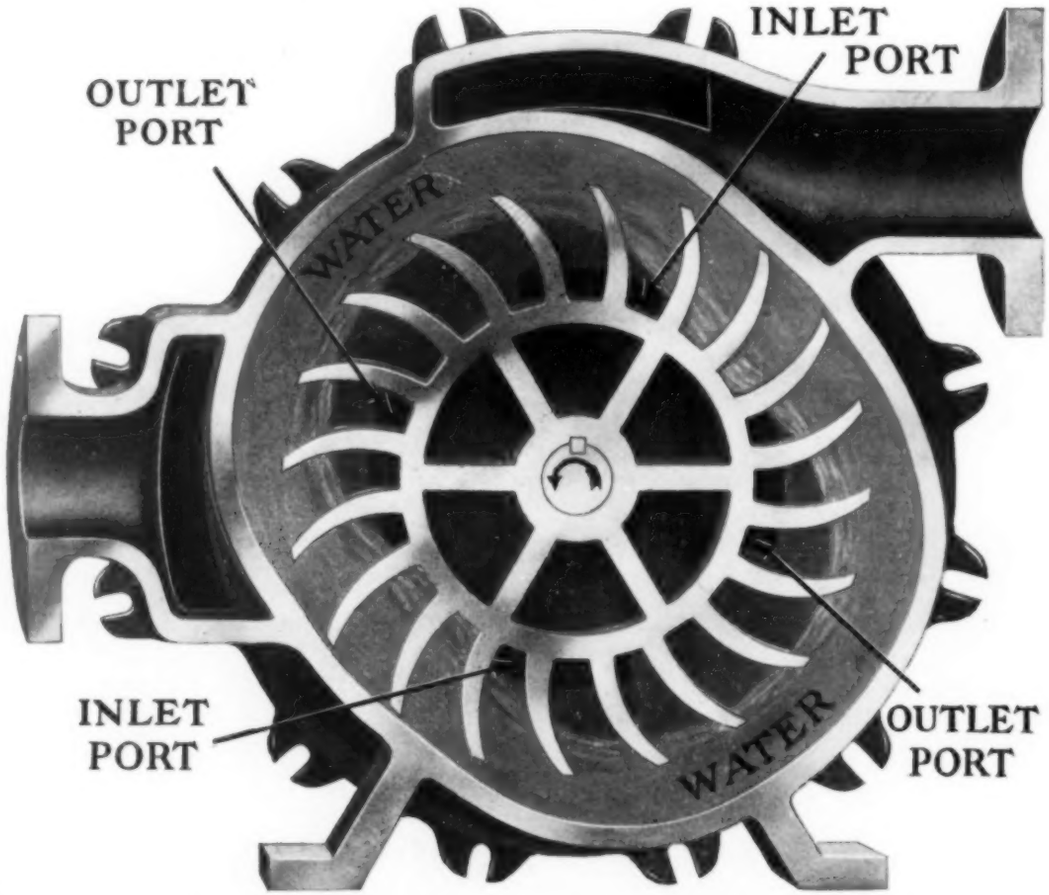


NASH HYDROTURBINE

THE SIMPLEST AIR COMPRESSOR AND VACUUM PUMP



Principle of Operation

The water revolves with the rotor but follows the elliptical casing due to centrifugal force. Twice in a revolution the water alternately recedes from and reënters the rotor.

The Water Acting as a Piston Compresses the Air.

The Hydroturbine contains but one moving part, the rotor, which is cast in one piece, with blades reinforced by shrouds cast integral. The rotor is firmly keyed to a substantial shaft which rotates on heavy annular ball bearings mounted outside of casing. The air is thoroughly washed during compression and contains no oil, as the Hydroturbine requires no interior lubrication. Air is delivered by the Hydroturbine without pulsation.

Various liquids may be utilized as the displacing medium, in connection with the handling of chemical gases.

See these pumps in operation at our exhibit, where Nash Engineers will be pleased to explain their application to your problems.

Booths 244 and 245 Mezzanine Floor

NASH ENGINEERING COMPANY

SO. NORWALK, CONN., U. S. A.

Published SEMI-MONTHLY *on the* FIRST *and* FIFTEENTH *of each month*

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Nec Timeo
Nec Sperno

THE rumor mill is always interesting and sometimes it is instructive. The latest, in regard to dyes, comes from a man who knows somebody in Switzerland who knows somebody in Sweden who knows somebody in Holland, and is as follows: After the war the Germans do not anticipate a great export of German dyestuffs and their best colors are not to be exported. Those already known are to be sold in competition against the products of other countries as opportunity offers, but the mysterious new ones, those with unheard of tinctorial power and endurance and brilliancy, are to be under embargo for the benefit of German textile manufacturers. They are alleged to have such amazing lustre and bloom and glow and to be so fast against the sun and wash and bleach and wear and mud and all the vicissitudes of tinctorial life that the dyes in present use will be wretched stains in comparison. We are not informed whether those with the photo-chemical properties dreamed of by Professor Ciamician, which shimmer with untold radiance as the fabrics colored with them are brought to the light, are included. He imagined a beautiful woman enrobed in such stuffs entering a brilliantly lighted room and growing more resplendent as she approaches. We might as well add this invention to the rumor to give it body and increase its stimulus.

Time has taught us that the composite German under his Prussian constitution does not play a gentleman's game in world affairs, and that if he is carefully observed and all cheating privileges are denied him, his boasting is vain and in point of ability he is no better than the other players.

In making dyes and intermediates he did get ahead of us, and his commercial methods were those of the card sharper's green table to which he so persistently invites his enemies to meet him and which his enemies so persistently decline to do. According to the rumor of the long journey, his present attitude in insisting upon the return of his colonies is to assure himself of a supply of cotton and wool (*sic*) so as to beat the world in textiles. Flax may be obtained from Russia and worked up in conquered Belgium and within the Vaterland, being provided for by the treaty made over the green table at Brest-Litovsk. For the past four years the things he has held sacred have been few in number. Lately he has added this treaty to his collection of sacred things and yet, somehow, it does not convince us of his spiritual growth.

He is a great hunter for vulnerable spots and vulnerable persons and as soon as he finds a foreigner who looks crooked or weak or mentally crippled with vanity, he approaches him with proffers of friendship and profit.

As he finds American men indisposed to accept his advances it will be no more than natural for him to appeal to American women, especially those of fashion and wealth, to buy his wares. And no matter how unwarranted the rumor as we have related it may be, we may be sure that in some way or other, he will make an ardent appeal to American women with proffers of finery after the war. The love of dress is strong among them and neither voting privileges nor political preferences are likely to overcome it. The dye industry is, in great measure, built upon it.

While the composite German is not a person of good taste, like the composite Frenchman, he is remarkable in imitation and in development of the ideas of others. Until he grows rich he is exceedingly industrious. When he has to root deep or die, he roots deep. And we may well conclude that in research in relation to dyes and textiles, he will continue to preserve. We have excellent research laboratories in connection with our leading dye-manufacturing establishments. We believe that in their facilities they are equal to those of the leading German works in time of peace and, as likely as not, somewhat better. In imagination and in chemical understanding we believe the great American laboratories to be as well if not better manned. On the other hand, we doubt if they have the many university connections that some German concerns enjoyed and used, and the American laboratories have devoted most of their time to the herculean tasks of the development of unfamiliar processes and to the achievements of quality and yield. The chances are that just now, the Germans are ahead of us in novelties. It is well to bear this in mind anyway, and to remember that as soon as we grow scornful and think they are not to be feared any more, they will rise again. At the dye symposium at the Cleveland meeting of the American Chemical Society, the chairman, Mr. R. Norris Shreve, pointed out that the American color industry cannot endure without constant and intense research—in fact, he declared that without it we are bound to fail. We cannot give too much emphasis to this wise statement, which is the very point we have been trying to illustrate.

Another report that comes by less roundabout way is that in the amalgamation of the German dye industries, the Verwaltungsrat or controlling body is made up of technically trained men. The selling staff tells what can be sold and is to report on markets while the financial advisors determine gross expenditures, but control will rest with men who do not require a treatise on chemistry to make them understand proposed measures. If they have their entire dye industry in such hands they are ahead of us in this respect. We do not want to harp on this subject too often but we are firmly convinced that the weak spot in far too many American industrial concerns is in the board of directors. A man does not think that he is entitled to sit on the board of directors of a bank because he deposits his money there, and no more should he presume to inflict his ignorance upon a manufacturing establishment because he has some money invested in it. Shareholders should be represented on boards by the co-operation and wisdom and understanding of the whole body and not by the ignorant and incompetent guardians of special interests. Such men cannot possibly understand technical problems as they arise, and they cannot judge properly when to say yes and when to say no. It is high time to go through some boards of directors of chemical organizations with a fine-tooth comb to remove the dandruff.

Sulphuric Acid

A Martial Indicator

High-school chemistry text-books of twenty-five and thirty years ago contained a statement regarding sulphuric acid that always held the attention of the student even though it was beyond his comprehension. This was to the effect that the consumption of sulphuric acid is an index of the degree of civilization of a people. The reader probably will recall earlier or later statements to the same effect, having reference, of course, to the peaceful pursuits and industries of a nation. But what of sulphuric acid in war? Of what is its consumption then an index? Perhaps a terse answer would be: Of the intensity and magnitude of the military operations.

In 1917 our production was over 7,000,000 tons, nearly twice that of 1913 which may be taken as a normal pre-war year. Plainly the consumption of sulphuric acid is an index of something more than the civilization of a people.

Additional information of unusual interest is contained in the report of the U. S. Geological Survey on the production of sulphuric acid in 1917. Compared with 1913, the last normal year prior to the war, the production for 1917 was nearly twice as great, the increase occurring in the acids of strengths higher than 66 degrees Baumé. The significance of this is, of course, that acid of high strength enters into the manufacture of munitions of war, and with the nations of the world straining every resource at their command for the production of munitions at a high rate of speed and in hitherto unheard-of quantities, sulphuric acid production increased out of all proportion to peace-time outputs.

The dislocation of world trade and commerce by the war had its effect on sulphuric acid production, and for a time the uncertainty of a ready supply of sulphur was disquieting. Wholly unmindful of the machinations of the Potsdam gang, our sulphuric acid industry had been built up largely on the use of pyrites imported from Spain. Thus in 1912 we imported 970,785 long tons of pyrites containing more than 25 per cent of sulphur; in 1913, 850,592 tons; in 1914, 1,026,617. This high mark was followed by a slump in 1915 to 964,634 tons; and this, in turn, by a rise to 1,244,662 tons. In 1917 the imports fell off to the level of 1912 and 1915, amounting to only 967,340 tons.

With these disturbances it became necessary to readjust our industry to the use of domestic sulphur-bearing materials and in some cases to remodel plants to burn native sulphur in place of pyrites. There was a wide search for sulphur-bearing ores throughout the United States, and while it can be said that we have ample supplies of sulphur-bearing ores, this does not easily solve the problem of acid production. There has been reluctance to use certain ores, notably pyrrhotite, on account of the changes in practice which would be involved. Regardless of these difficulties, however, and the further problems of labor shortage and transportation, the industry is serving the warring nations with remarkable ability.

Sidelights on the Exposition

IT IS not only little children that want to see the wheels go round. The crowd likes to see things moving, provided something is happening in the process that those who compose the crowd can understand. A series of mystical prayer wheels, whirling about as is shown in the last act of The Magic Flute will cause anybody to stop and look as he passes by, but there needs to be some sort of an answer to the prayer going on to attract his sustained attention. A number of exhibitors have availed themselves of this disposition of persons to close in and see what is happening, and with considerable success. Thus visitors hover about a Dorr thickener and a Benith rotary filter in operation. They watch a pretty glass-and-brass model of a Ruggles-Coles direct-heat dryer turning about with material passing through it. They gather around a furnace burning powdered coal designed to heat apartment houses and office buildings shown by the Powdered Coal Engineering and Equipment Co. and about the dyeing operations in process in the booth of the National Aniline & Chemical Co. We mention these merely as examples, for there are many others.

* * *

The Solvay Process Co. has a thrilling panorama of a war scene. There are aëroplanes in the air, a burning village in the distance, artillery at work, tanks big and little going out, wounded men on stretchers before a dressing station, ambulances going and coming, a Y. M. C. A. hut—in short, nearly everything except the smell of battle, the noise, the misery and the hate. Then hung up above and at the sides of the picture are Solvay products and the uses to which they are put in war, all clearly indicated. The Semet-Solvay Co. also shows a connected display of coal and its products while their relations to each other are indicated by silk ribbons which bind them together.

* * *

It was a happy idea to mark exhibits of things made in the U. S. for the first time since the war began with a bull's eye. We shall make note of some of these occasionally as we proceed, but we give notice here and now that these comments are in no sense a catalogue of the show. They are merely side lights and we deny all obligation to observe a proper sense of proportion.

* * *

The Barrett Company displays a long list of products marked with the eye that sees red. These include anthracene, carbazol, orthocresol, meta-paracresol, various forms of paracumaron resins, resorcine, pyridine and others. The same company shows a remarkably interesting coal-tar tree of unusual detail, in which not only the names of a large number of coal-tar derivatives are given but also a sample bottle of each product, displayed in its proper place.

* * *

The Anaconda Copper Mining Co. has, of course, a considerable display of copper and we also find, side by side, samples of tellurium and selenium. About an equal amount of each of these cousins to sulphur is shown, but we doubt if any copper refiner maintains the same sentiments towards both of them. We understand that there is almost a greater demand for selenium than the market can meet whereas tellurium is still looking for a job. Here's a good chance for a doctor's degree and a fortune for some aspiring young research chemist, in the discovery of an industrial use for tellurium. The principal application of selenium is said to be in correcting the color in glass, owing to its red effect. At all events that is one large use. We heard a rumor that some of it goes into the dyestuff industry, but we are addicted to doubt in this direction. On the other hand, we throw out a suggestion free of charge to the prospective research wallah in tellurium: Why not make tellurium dyes in the place of sulphur colors? Tellurium is cheap and it has a grand heft. Moreover, next to body and bloom in color, textile people are often made glad by the excess weight that accrues to fabrics in the dye bath.

It does not take much of a strain upon the memory to recall the time when certain wise men refused to consider electrolytic zinc with that quality of faith which is the substance of things hoped for. But here it is, in handsome quantities, 99.9 per cent pure.

* * *

Major F. E. Breithut, who has Uncle Sam's exhibit in charge is connected with the personnel division of the Chemical Warfare Service. He tells us that the immediate, return-mail responses to the questionnaire lately sent to chemists over his name, have numbered over 7000.

* * *

The face-piece of the captured German gas-mask shown in the exhibit of the Chemical Warfare Service is made of softened leather instead of rubber, for obvious reasons. The edges are of cotton fabric, covered with some sort of varnish. The tubes are made of similar cloth and inclose coiled springs of metal, to give them the necessary elastic effect. It is a clever makeshift.

* * *

Corporal Charles F. Roth of the Chemical Warfare Service is, in private life, joint manager of the Exposition with Mr. F. W. Payne, and Secretary of the New York Section of the American Chemical Society. It isn't letting the cat out of the bag to say that when, under the selective draft, Mr. Roth was called to the colors, it became necessary for his associate, Mr. Payne, to cinch up his belt and, metaporically speaking, spit on his hands for the job ahead of him. A year ago, in the comedy of circumstances, the manager of the Exposition would, as likely as not, have been set to work to peel potatoes in some camp while the authorities were in despair to know the very things in which this cheerful corporal is peculiarly well posted: as to who makes what. A month or so ago, however, when we saw him for nearly the first time in uniform since he wore a little sailor suit as an angel child, he had just been transferred to the Chemical Warfare Service, and he was working like a beaver to speed up production. A modest, gentle-spoken soul is the corporal, far rather disposed to sweet reasonableness than to the sway of might. He is not of the shouting kind. His name has a German turn to it, but that comes from earlier generations, and we can no more imagine him making Prussian noises than we can imagine him mutilating children.

The Direct Determination of Sulphur Dioxide in Burner Gases

By F. N. Williams

WITH the urgent need of higher efficiency and conservation in the use of sulphur, proper burner regulation and furnace control are matters of the utmost importance.

In order to obtain the maximum yield of sulphur dioxide, either from the direct combustion of sulphur or pyrites, regular analysis of the burner gases is most imperative. In this way only can the proper air regulation and burner temperature be obtained. There are two general methods for the determination of sulphur dioxide. The first is by means of an apparatus of the Reinsch type, in which a measured volume of the gas is passed through suitable absorption bottles containing standard iodine solution. The end point is determined either by the disappearance of the color of the iodine or by the titration of the excess of iodine with sodium thiosulphite. In the writer's experience this method is open to several very serious objections, as follows: It is slow and tedious, completeness of absorption is always uncertain, as there is no way of checking back the sample to see if all of the sulphur dioxide has been absorbed. The apparent volume of the gas measured is also uncertain, due to difficulty in maintaining constant temperature and pressure conditions. Further, even the calculation of the percentage of sulphur dioxide is based upon the use of impirical factors which may or may not apply to the conditions of the analysis. The direct method for the determination of sulphur dioxide, as well as all other chemical gases, such as chlorine, hydrochloric acid gas, oxygen, carbon, monoxide and others is not open to the above objection, but on the other hand, is rapid, accurate and reliable.

With the instrument illustrated in Fig. 1, an accurate determination of the percentage of any of the above gases may be made easily in less than five minutes, including the time required for taking the sample.

This is a modification of the Orsat, combining the many good qualities of this instrument with the elimination of its faults. Its manipulation is simple and easily mastered, even by those not experienced in chemical laboratory work. It is portable and selfcontained, easily carried to any part of the plant.

To make an analysis, it is only necessary to set or hang up the instrument near the source of supply of gas to be tested and connect sample tube, after which a measured volume of 100 cc. of sample is taken into the instrument when, by raising the leveling bottle, the sample is passed through the absorbing solution in the special bubbling pipette, after which the unabsorbed residue is returned to the burette and measured. The percentage of gas absorbed is read off directly on the graduated measuring burette. The operation may be repeated and if the reading is found to be constant, completeness of absorbtion is certain.

In the proper regulation of sulphur burners, a determination of the amount of air or oxygen in the combustion gases is most important. The same instrument is used for the determination of oxygen in the damper regulation, as well as for the detection of air leaks in the line.

In addition to the many advantages of simplicity, accuracy and rapidity, the Williams improved gas apparatus has the widest possible range of adaptability. The same instrument may be used for flue-gas analysis in the boiler room, the determination of sulphur dioxide, chlorine, hydrochloric acid and other corrosive gases, as well as for the complete analysis of combustible, illuminating and fuel gases.

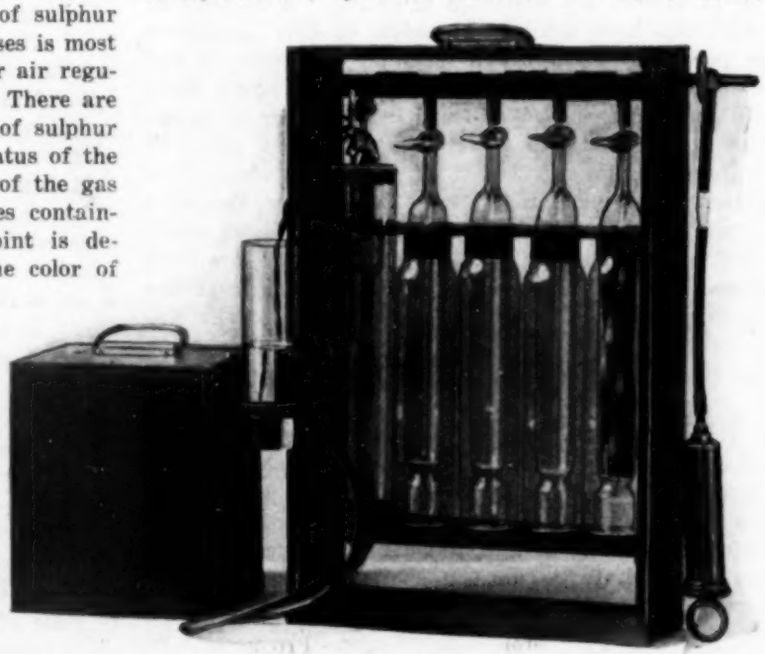


FIG. 1.—WILLIAMS GAS ANALYSIS APPARATUS

Gas Analysis With Very Small Quantities

An apparatus is described by means of which very small quantities of gaseous mixtures may be analyzed, and in which, during the analysis, the gas comes in contact only with glass, platinum, and mercury. The gas is freed from moisture and carbon dioxide by cooling with a mixture of solid carbon dioxide and alcohol and with liquid air respectively, then burnt by means of a platinum spiral, after addition of oxygen or of hydrogen if necessary; excess of unburnt oxygen is determined by adding hydrogen and again burning, the use of an iron spiral having proved unsatisfactory. A capillary tube is used for the combustion, and if this be effected at a low pressure combination of nitrogen and oxygen and of oxygen and mercury vapor may occur and lead to incorrect results. The use of automatic pumps of the Sprengel type for transferring the gas from one part of the apparatus to another is also inadmissible, as gas occluded by the mercury is liberated when the pressure falls very low and may pass into the measuring tube. The use of Töpler pumps overcomes this difficulty. Results obtained with quantities of gas of the order of 10 cu.mm. are given. In the case of a mixture of H_2 26.7, CO 22.7, CH_4 22.9, and N_2 27 per cent, the values found were H_2 26.4, CO 21.1, CH_4 24.2, and N_2 28.3 per cent.—*Z. anal. Chem.*

Program of Fourth National Exposition of Chemical Industries

Grand Central Palace, New York
Week of Sept. 23, 1918

The addresses comprise a series of symposiums devoted to a consideration of

THE DEVELOPMENT OF CHEMICAL INDUSTRIES IN THE UNITED STATES, NOTABLY SINCE JULY, 1914.

Wednesday, September 25th.

Afternoon (2:30 P.M.) Symposium: POTASH.

C. A. HIGGINS (Hercules Powder Co.), Recovery of Potash from Kelp.

LINN BRADLEY (Research Corporation), Recovery of Potash from Iron Blast Furnaces and Cement Kilns by Electrical Precipitation.

JOHN W. HORNSEY, Potash from Desert Lakes and Alunite.

ALFRED DE ROPP, JR. (American Trona Co.), Potash from Searles Lake.

Evening (8 P.M.) Motion Pictures.

Electrical Precipitation of Potash from Cement Dust (Research Corporation).

*Colloid Chemistry.

The Operation of a By-Products Coke Plant (2 reels). (H. Koppers Co.)

Moving a Forest to France (4 reels). (Southern Pine Association.)

The Story of Potash—Production at Searles Lake.

Thursday, September 26th.

Afternoon (2:30 P.M.) Meeting: AMERICAN CERAMIC SOCIETY—Chairman, L. E. Barringer.

L. E. BARRINGER—Manufacture of Electrical Porcelain (Illustrated, Motion Picture).

A. V. BLEININGER—Recent Developments in the Ceramic Industries.

H. RIES—American Clays.

F. A. WHITAKER—Manufacture of Stoneware (Illustrated, Slides).

J. B. SHAW—Fuel Conservation.

S. C. LINBARGER—Carborundum Refractories.

These will be followed by motion pictures as designated for the evening program.

Evening (8 P.M.) Motion Pictures.

THE CERAMIC INDUSTRIES

Glass Making (Corning Glass Co.)

†The Making of Cut Glass.

Manufacture of Electrical Porcelain (General Electric Co.).

†The Making of Pottery.

(9 P.M.) Speakers:

J. A. SWITZER (University of Tennessee), Waterpower Potentialities of East Tennessee.

HERBERT C. HENEGAR (American Zinc Co.), Mineral Resources of East Tennessee.

From A to Z with the Exhibitors

IN THIS and succeeding daily issues of CHEMICAL & METALLURGICAL ENGINEERING published during the Chemical Exposition will be found brief descriptions of more than 300 exhibits. In order that the record might show the progress that has been made since we entered the war, we have asked the exhibitors to inform us which of their products have been made in this country for the first time since the war began. At the exposition itself the visitor will notice many chemical products marked with a bull's-eye as the distinguishing mark of such progress.

PAUL O. ABBE: Single specimen, assay and jar mills, direct-connected to motors. Duodecuple assay mill, improved rotary cutter, hammer mill, and pony mixer. In charge of exhibit: Mr. ABBE, Miss LAMA.

ABBE ENGINEERING CO.: Patented pulverizing and grinding machinery; laboratory grinding mills; rotary cutters. Special laboratory mills for pulverizing chemicals have been developed since we entered the war. In charge of exhibit: H. F. KLEINFELDT, H. C. RUSS, W. S. RANSOM.

AMERICAN BLAUGAS CORPORATION: Blaugas for operation of laboratory gas appliances and for industrial heating purposes. Dri-Gas for welding, cutting, and brazing. The latter is a new product since the United States entered the war. In charge of exhibit: G. E. BEVES, T. F. HINTZE.

AMERICAN CERAMIC SOCIETY: Charts showing in simplest possible form the size and importance of the ceramic industries both in themselves and in relation to other well known industries.

AMERICAN CHEMICAL & MFG. CO.: Chemical oils, lacquer, technical coatings, acid-proofing and waterproofing, protective coatings for airplanes, submarines, auto-cars, steel cars, steel construction, etc. The new developments since we entered the war are coatings for concrete ship bottoms; anti-fouling ship-bottom paint; pyroxilin coatings for airplanes. In charge of exhibit: W. M. VAN OSTROM, CHAS. D. STILLMAN, F. E. SINCERE, R. C. DEDERICK, E. K. CHAFFEY, F. K. LOCKHART.

AMERICAN CYANAMID CO.: A complete set of cyanamid products: cyanamid, aqua ammonia, nitric acid, ammonium nitrate, sulphate of ammonia, ammo-phos, urea, dicyandiamid, cyanide and Florida pebble phosphate. Cyanamid is the only one of these products produced prior to the war; all others have been developed since 1914. A series of samples will show the steps in the manufacture of cyanamid. Urea is a product that has come into considerable prominence on account of its use as an acid absorbent in nitrocellulose and lacquer used on the fabric of aeroplane wings. Its presence in the dope neutralizes the slight traces of nitric acid that slowly develop and which would otherwise attack and weaken the fabric. Photographs and charts will complete the exhibit. In charge of exhibit: E. J. PRANKE.

AMERICAN ELECTROCHEMICAL SOCIETY. Heavy Browning machine gun in the manufacture of which artificial abrasives are used for between sixty and seventy grinding operations, chrome-carbon alloy steel, manufactured in the electric furnace, is used for the barrel; 8-in. army shell and 6-in. shell, trench hat,

EVERLASTING
EVERLASTING
VALVE
COMPANY
NEW YORK

list of processes used extensively in modern warfare; collection of products of electrolytic cell and of the electric furnace, together with photographs of waterpower devices. In charge of exhibit: CARL G. SCHLUEDERBERG.

AMERICAN ANILINE PRODUCTS, INC. A practical exhibit of dyestuffs.

AMERICAN KRON SCALE CO.: Kron automatic dial scale, 1000 lb. capacity, with platform 46 x 36 in., equipped with tare beam for automatic deduction of tare weight. In charge of exhibit: E. OHNELL.

AMERICAN-LA FRANCE FIRE ENGINE CO., INC.: Respirators for protection against dust and fumes. Filter masks for various dust and poisonous fumes. The latter article is a new development following somewhat the idea of the army trench mask.

THE AMERICAN METAL COMPANY, LTD. The following products will be exhibited: Arsenic oxide, coal, copper, copper sulphate, ferroalloys, gold, lead and antimonial lead, molybdenite, muriatic acid, pyrites, salt cake, silver, spelter, sulphuric acid, tin, tungsten concentrates, zinc dust, oxide and sulphate. A special feature is the production of molybdenum concentrates on a large scale. In charge of exhibit: H. M. BURKEY.

AMERICAN METER CO.: Apparatus appertaining to the correct measurement and testing of gas, including calorimeters, siphon gages, sulphur tests, wet test meters, diaphragm dry meters, iron case meters, Tobey meters. The central feature of the exhibit is the calorimeter. In charge of exhibit: ERNEST KERSTEIN.

AMERICAN PIPE BENDING MACHINE CO.: "Wonder" pipe bending machines in two models. In charge of exhibit: E. T. BLAKE, GEO. W. GRAHAM.

AMERICAN WATER SOFTENER CO.: "Delcalso" zeolitic water softener. Glass apparatus will be in operation in which interested persons can soften any samples of water which they may bring with them. This device has been developed and brought out since we entered the war. In charge of exhibit: R. W. CONRAD.

ANALINE DYES AND CHEMICALS, INC.: Dyestuffs, intermediates and chemicals which are the products of the company's various factories. Many of the dyes and intermediates have been developed since 1914. In charge of exhibits: W. H. VAN WINCKEL.

APEX CHEMICAL CO., INC.: Chemical products used in the textile, leather and allied industries. Since the United States entered the war this company has produced the following chemicals: antimonelle, a substitute for tartar emetic; orthophene, for freeing lime from hides; lactic and formic acids. In charge of exhibit: Dr. S. M. HERMANN, HUGO HELBURN, CHARLES D. RASENBERGER, NELSON CRAIG.

ARKELL SAFETY BAG CO.: "Arksafe" elastic paper lining for barrels, drums, boxes and bags. These linings prevent the sifting of powdered materials from packages and keep the contents clean and dry. In charge of exhibit: C. J. MORALES.

ARNOLD HOFFMAN & CO., INC.: Joint exhibit with Mathieson Alkali Works, Inc., and Nitrogen Products Company. Exhibit of bleaching powders, liquid chlorine, caustic soda, soda ash, sodium bicarbonate and modified sodas, sodium cyanide, red and yellow prussiate of soda and anhydrous ammonia. Pigment colors, gums, dextrine, oils, softeners and textile finishing materials. In charge of exhibit: A. L. HAYES.

BARIO METAL CORP.: Bario metals used in some cases as a substitute for platinum. Electric resistance furnaces. Both the metal and the furnace have been developed since we entered the war. In charge of exhibit: PAUL DEMILES.

BAYONNE CASTING CO.: Monel-metal castings, nickel castings and hot-rolled monel-metal rod. Fabricated monel-metal shapes. In charge of exhibit: H. F. CHASE.

BAUSCH & LOMB OPTICAL CO. A complete phopomicrographic outfit for metallographic work, suitable for work with high magnifications; a complete compound metallurgical microscope and smaller accessories. In charge of exhibition: R. C. DEAN.

BEACH-RUSS CO.: Patented rotary high-vacuum pumps, rotary pressure blowers. Extra high-vacuum pumps for chemical plants have been developed since we entered the war. In charge of exhibit: H. C. RUSS, A. T. BEACH, C. A. BEACH.

CHRISTIAN BECKER, INC.: Becker analytical balances, including chainomatic analytical balance and chainomatic specific gravity balance. Torsion balances. The chainomatic balances are new developments since we entered the war. In charge of exhibits: S. W. HESS, Miss C. L. BECKER, A. T. MILLROY.

BECKLEY PERFORATING CO.: Perforated metal products and miniature reproductions of tanks and special shapes in sheet metal which have been furnished to various chemical industries. Tanks and special shapes are electrically welded. In charge of exhibit: ALLEN L. PRICE.

BETHLEHEM FOUNDRY & MACHINE CO.: Castings made of corrosiron. It has been necessary for the present to discontinue the manufacture of tantiron castings owing to shipping difficulties. New developments since the war began have been along the line of confidential work for clients. In charge of exhibit: J. GEORGE LEHMAN, R. E. WILBUR, GEORGE ETTELE.

BOYER OIL CO., INC.: Oils and fats, including castor, cocoanut, corn, cottonseed, cod, linseed, mustard, olive, palm, peanut, rapeseed, sesame, soyabean, walnut and whale oils. In charge of exhibit: Mr. KIENLE.

BRISTOL CO.: Recording and indicating instruments for pressure, temperature and electricity.

BUTTERWORTH - JUDSON CORP.: Dyestuffs, intermediates, heavy acids, salt cake, nitre cake, lithopone.

A. M. BYERS CO.: Genuine wrought iron pipe, showing the method of marking every length with name and year of manufacture. Samples of pipe taken from service in an effort to determine relative life of iron and steel pipe. In charge of exhibit: R. W. KENNEY, N. BOLAND.

JOHN CAMPBELL CO.: Aniline colors and coal tar products. Since the United States entered the war this company has produced a full line of direct, basic chrome, acid and sulphur colors sold under trademarked American names. The line is being constantly enlarged. In charge of exhibit: GEORGE H. WHALEY.

CANADA CARBIDE CO., LTD.: Samples of domestic and export sizes of carbide; domestic and export packages; projection lantern and colored slide. In charge of exhibit: H. E. MUSSETT.

CANADIAN CHEMICAL JOURNAL: A representative will be in charge to give information on Canada, statistics

of Canadian products, especially those relating to the chemical and metallurgical industries. In charge of exhibits: E. B. BIGGAR, L. E. WESTMAN.

CARBORUNDUM CO.: Carborundum protection pyrometer tubes, carborundum fire sand, silicon metal. Carborundum resistance rods for use as lightning arresters or similar work where a static resistance only is required. Miscellaneous exhibits of small refractories made from silfrax. High grade fire brick, carbofrax and refrax.

CARRIER ENGINEERING CORP.: Model Carrier system of air-conditioning in operation. Samples of materials in the manufacture of which the Carrier system plays an important part. In charge of exhibit: J. I. LYLE, E. P. HECKEL, L. L. LEWIS, M. S. SMITH, GEORGE SCHMIDT.

CELLULOID ZAPON COMPANY. Lacquer enamels and varnishes, bronze liquids, amyl acetate, soluble cotton.

CENTRAL DYESTUFF & CHEMICAL CO. Coal-tar colors and intermediates. In charge of exhibit: M. UNDERLEIDER, A. F. WIEHL, E. MONTALENT.

CENTRAL SCIENTIFIC CO.: Laboratory specialists; de Khotinsky constant temperature apparatus, Parr Calorimeters; balances and weights; electrical instruments. Since the United States entered the war the company has produced special analytical balances, vacuum pumps, stills and rotary blowers. In charge of exhibit: S. L. REDMAN.

CHEMICAL CO. OF AMERICA, INC.: Intermediates for dyestuff manufacture, in addition to which will be featured a khaki color. In charge of exhibit: Messrs. KENDALL and CHRIST.

CHEMICAL CONSTRUCTION CO.: Blueprints showing the installation of different kinds of acid plants constructed by the company. A model acid-proof masonry tower showing detailed interior construction. Samples of acid-proof brick and acid-proof cement joints. The acid-proof masonry has played an important part in the construction of government nitrate plants throughout the country. The spiral chemical packing rings for use in absorption towers have been produced in excess of 100,000,000 this year. In charge of exhibit: R. L. RUTZLER, T. C. OLIVER.

CHEMICAL WARFARE SERVICE, NATIONAL ARMY: Information bureau regarding industrial relations and university relations. Exhibit of gas masks by Gas Defense Division. Exhibit of Gas Offense and Research Divisions. In charge of exhibit: Maj. F. E. BREITHUT.

CLINCHFIELD PRODUCTS CORPORATION: Full line of products, including sodium sulphide, barium carbonate, barium chloride, barium sulphate, barium sulphide Glauber's salts, and ground feldspar. In charge of exhibit: J. G. HARRIS.

CRANE CO.: A line of valves, fittings, and specialties for steam-power, heating and process work and for water, gas, and chemical purposes. The feature of the exhibit will be a complete steam plant in actual operation, demonstrating the company's products but particularly the boiler-feeding system. In charge of exhibit: W. L. OSWALD, WM. FINK.

CRANE PACKING CO.: "John Crane" babbitt foil metallic packing, which is made of very thin sheets of babbitt foil, spirally wound to allow of the greatest amount of compression and flexibility. This material is specially applicable to use in the chemical industry. Centrifugal water-pump sets of packing will be shown, and asbestos copper foil wrapped packing for superheated service. In charge of exhibit: F. E. PAYNE, JULIAN N. WALTON.

CRESCENT COLOR & CHEMICAL WORKS, INC.: An exhibit of dry and pulp colors. In charge of exhibit: N. A. MCMANUS, JAMES MURRAY, JOHN GLEDHILL, V. J. CHARTRAND.

CRESCENT INK & COLOR CO., INC.: An exhibit of the manufacture of ink. In charge of exhibit: N. A. MCMANUS, JOSEPH M. PETRY, WALTER A. CONLAN.

CROMOS CHEMICAL CO., INC.: A display of benzoic acid, benzoate of soda and benzyl chloride. In charge of exhibit: I. MARK, J. H. LYONS.

J. H. DAY CO.: Mixing machines for handling powders, liquids, and pasty materials. In charge of exhibit: R. W. WALLACE.

DENVER FIRE CLAY CO.: Laboratory and plant specialties including furnaces, crushers, samplers, screens, fire-clay products, electroscope equipment for testing radioactivity, oil and gas burners. In charge of exhibit: J. S. KING, E. R. HARRIS.

DETROIT RANGE BOILER CO.: "Perfect" metal bilge barrel and drum, used as steel containers in the chemical industry. The tight barrel is suitable for handling liquids. The removable-head barrel can be used for powders, semi-solids, heavy liquids, and other products the emptying of which requires the entire head to be taken out of the barrel. In charge of exhibit: W. B. GOGGARD.

J. P. DEVINE CO.: Vacuum drying chambers of several sizes made up in complete installation form, with all necessary fittings, mountings and auxiliaries, a condenser and a rotary valve vacuum pump; a high-pressure cast steel autoclave; copper distilled-water outfits capable of delivering 5 gallons of pure distilled water per hour; a double-effect vacuum evaporator suited for the experimental and research laboratory; some chemical equipment and a display of photographs. In charge of exhibit: J. P. DEVINE, NATHAN OWITZ, L. W. TREICHLER, A. D. RENNER, A. M. FRANTZ, E. H. MILLER.

DIAMOND STATE FIBRE CO.: Diamond fibre products including receptacles for mills, fibre sheets, rods, and tubes employed in the electrical industry. Features developed since the war are red spinning parchment, Diamond-F food-protective papers. In charge of exhibit: T. M. BOGERT.

DORR CO.: The Dorr agitator, Dorr bowl classifier, Dorr tray thickener, and Dorrco pump in closed circuit, demonstrating their various functions in a number of different chemical processes. A pulp containing different materials will be mixed in the agitator, separated in the classifier, thickened and dewatered in the thickener, and handled by means of the pump. This equipment has recently entered into the construction of several explosive plants built for the Government. In charge of exhibit: H. N. SPICER.

DYE PRODUCTS AND CHEMICAL CO., INC.: A display of dyestuffs and intermediates used for the manufacture of khaki dyestuffs for U. S. Government uniforms showing the various steps necessary in the manufacture of these materials. In charge of exhibit: SIDNEY SIMON, L. M. LOWENTHAL, CLARENCE K. SIMON.

Development in Nitric Acid Manufacture in the United States Since 1914*

Nine-Fold Increase in Production Since War Began—Ammonia Oxidation Process May Be Greatest Source of Production After the War—Data on Catalyzer Efficiency

By E. J. PRANKE

THE production of nitric acid in 1914, according to the census of manufacturers, was 78,589 tons of nitric acid of average strength, and 112,124 tons of mixed acid. According to other data given in the census, these figures represent about 89,000 tons of 100 per cent nitric acid. All of this acid was produced from nitrate of soda, consuming about 160,000 tons of nitrate. The pre-war importation of nitrate of soda amounted to about 560,000 tons per annum; hence the normal consumption for purposes other than the manufacture of nitric acid was about 400,000 tons.

The present rate of importation is about 1,600,000 tons of nitrate per annum. Since very little is going into storage and the total consumption for purposes other than nitric acid maufacture has increased but slightly, if at all, it may be estimated that at least 1,000,000 tons of nitrate per annum is being converted into nitric acid at the present time. This is equivalent to 650,000 tons of 100 per cent nitric acid, of which nearly five-sixths is being used for the manufacture of military explosives.

Improvements In Recently Built Plants

The building of the new nitrates of soda acid plants has offered an excellent opportunity for the introduction of many improvements. The Dutch ovens under the retorts have been displaced by modern fire-boxes provided with a proper arch. This change has effected a saving in coal consumption of approximately 25 per cent. The chemical stoneware from the retorts to the condensers and the glass condenser tubes have been displaced by acid-proof, high-silica iron, such as duriron and tantiron. The volvic-ware saucers in the towers have also been displaced by acid-proof iron. The chemical ware from the condensers to the absorption towers, and the glass lines for circulation of acid at the sides and top of the towers, however, are retained. The absorption tower capacity has been increased about 40 per cent by the addition of more towers. Spiral rings for tower packing have taken the place of the ordinary form of packing.

Important changes have also been made in operation. The average charge of 5000 pounds of nitrate per retort has been increased to about 7500 pounds. The retorts, instead of being operated in batches, are now operated in rotation. Instead of three runs per retort per day, the usual practice is now two runs per day. Temperatures are also controlled more carefully than in the past.

The result of these improvements is an increase in the amount of nitrogen recovered as acid from an average of from about 78 to 80 per cent to about 92 to 94 per cent of the nitrogen in the nitrate of soda. At the same time the labor requirement has been somewhat decreased.

A good beginning has also been made in the recovery of nitrose gases produced in the various nitration operations. In some of the systems that have been devised, as much as one-half of the fumes are being recovered. The collection of the gases from the many nitration units, however, is still a serious problem. While the aggregate amount of acid that is being recovered is large, it represents only a small fraction of the acid gases that are being wasted. In the ordinary nitration operation as carried out at present, about one-tenth or one-twelfth of the nitric acid is wasted in the wash waters, while on an average about one-eighth is lost as fumes, of which one-half is recovered in plants equipped with recovery systems.

Nitric acid by direct combustion of air by the arc process has not had any important development as yet in America. Three small plants, more or less on an experimental scale, have been built in the United States and operated for short periods. The production of these plants thus far has been negligible. The total annual capacity probably does not exceed two or three thousand tons of nitric acid per annum.

Nitric acid by the oxidation of ammonia has received a considerable and important development since the outbreak of the war. In 1914 there were no ammonia oxidation plants in this country. At the present time there are under construction ammonia oxidation plants with a capacity equal to about 225,000 tons of 100 per cent nitric acid per annum.

Development of Ammonia-Oxidation Plants

The first commercial-sized oxidation plant was established in July, 1916, at the Ammo-Phos Works of the American Cyanamid Company at Warners, New Jersey. Six catalyzer units were installed, each with a presumed capacity of 14 pounds nitric acid per hour. Improvements in the design of the catalyzer and in methods of operation have brought the capacity to over 40 pounds of nitric acid per hour. The catalyzer used is a single fine platinum gauze, with an area of about two square feet, electrically heated. Over a period of two years two of these units have supplied the nitric requirements of the 60,000-ton sulphuric acid chamber plant at this works. The ammonia is taken directly from cyanamide autoclaves producing about 30 tons of ammonia gas per day, used mainly for aqua ammonia manufacture. This plant has served for several months as a training school for the instruction of operatives for the Government cyanamide-nitrates plants. As an

*A paper read at the Fourth National Exposition of Chemical Industries, New York, Sept. 24, 1918.

example of the normal operation of the catalyzers on ammonia taken directly from the autoclave mains, Table I is quoted verbatim from the records for the week July 13 to 19, 1918. Each value is the average of determinations of two chemists working independently, with the exception of those marked (*) which are determinations of one chemist only.

TABLE I.—EFFICIENCY OF CATALYZERS IN AMMONIA OXIDATION

Catalyzer No. 5			Catalyzer No. 6		
Date	Time	Efficiency	Date	Time	Efficiency
July 13....	2.35 a.m.	96.2	July 13....	5.30 a.m.	90.0
	8.40 a.m.	98.5		1.10 p.m.	93.0
	5.20 p.m.	93.1		8.40 p.m.	93.4
	11.50 p.m.	96.0	" 14....	2.50 a.m.	93.0
" 14....	5.50 a.m.	97.4		1.00 p.m.	94.2
	5.10 p.m.	94.8		10.15 p.m.	93.2
" 15....	10.35 a.m.	95.0	" 15....	8.15 a.m.	95.6
	5.15 p.m.	95.8		2.30 p.m.	90.7
" 16....	1.50 a.m.	95.4		11.30 p.m.	92.6
	11.20 a.m.	95.4	" 16....	8.00 a.m.	90.0
" 17....	1.10 a.m.	92.1		8.25 p.m.	93.0*
	1.00 p.m.	92.3	" 17....	9.30 a.m.	90.9
" 18....	12.50 a.m.	93.4		8.20 p.m.	94.0
	10.55 a.m.	92.6	" 18....	7.40 a.m.	92.0
	8.00 p.m.	91.9		5.10 p.m.	91.6*
" 19....	9.50 a.m.	93.6	" 19....	4.15 a.m.	93.0
Average for the week		94.5	Average for the week		92.5

Muscle Shoals Plant in Operation November First

The cyanamide-nitrates plant at Muscle Shoals, Alabama, will use the electrically heated single-gauze catalyzer. It will produce approximately 90,000 tons of 100 per cent nitric acid per annum. The plant is expected to go into operation about November 1. The cyanamide-nitrates plant near Cincinnati, and the one near Toledo, Ohio, will also use the same process, each producing at one-half the above rate. They are expected to be in operation early next spring.

The Government experimental plant at Sheffield, Alabama, known as Nitrate Plant No. 1, which will make about 15,000 tons of nitric acid per annum, has adopted a non-electrically heated multiple screen, consisting of several layers of platinum gauze, welded together at points, and rolled into the form of a cylinder. The ammonia-air mixture flows outward through the screen at a rate several times as fast as with the electrically heated single screen. After the oxidation has been started by external application of heat the temperature is self-sustaining from the heat of reaction.

Negligible Consumption of Electric Energy

In view of the perfect control obtainable with electrical heating, the cost of the electric energy consumed, amounting to about one-third of one per cent of the present market value of the nitric acid, may be regarded as negligible. As to the single versus the multiple screen the efficiencies cited above as examples of normal operation of electrically heated single screens are believed to represent the highest standards yet attained in the practical operation of ammonia catalyzers.

It is understood that the Semet Solvay Company has an ammonia oxidation plant at Syracuse, New York, using the multiple screen without electrical heating. This plant is producing several tons $NaNO_3$ per day. Information regarding efficiencies is not available.

In addition to the plants above mentioned, the Navy Department decided about two months ago to build a plant at Indian Head, Maryland, for fixing nitrogen by the modified Haber Process used at Plant No. 1. All the ammonia produced will be oxidized to nitric acid, yielding about 30,000 tons per annum.

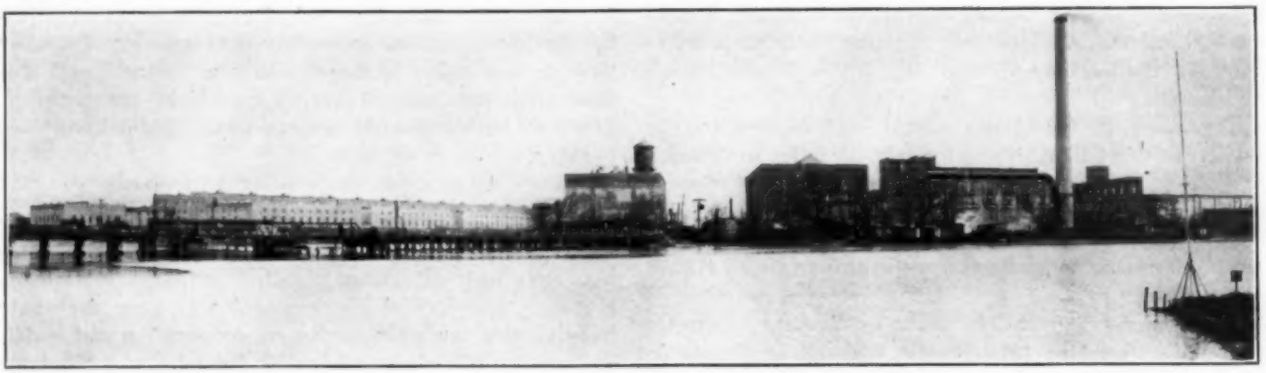
Considerable work is also being done on the use of catalyzers to hasten the conversion of the nitrose gases, obtained from the catalyzers, into nitric acid. The object is to reduce the amount of space required for reaction chambers. The experiments along this line show promise of early success.

Production Next Spring Will Be Nine Times Normal

The nitric acid producing rate in the spring of 1919 will be about 650,000 tons from nitrate of soda and about 225,000 tons by oxidation of ammonia obtained from the air, a total of $75,000 tons of 100 per cent nitric acid. This is about nine times the pre-war normal consumption. In 1914 the industrial explosives industry consumed about 50,000 tons per annum, while all other uses took only about 40,000 tons. The only notable increase in consuming ability since 1914, aside from military explosives, has been in the dye industry. In 1917 it was estimated that 30,000 tons of dyes were produced in America, equal to the total 1914 consumption. The production will probably increase somewhat further, but at most could hardly consume more than 30,000 or 40,000 tons of concentrated nitric acid. With a producing rate of 875,000 tons, and a consuming ability in peace times of 125,000 tons, or possibly 150,000 tons, it is evident that over four-fifths of the nitric acid producing capacity will have to be shut down as soon as peace conditions are established.

Ammonia-Oxidation Method May Be Principal Source

The successful development of the ammonia oxidation process raises the question whether this may not become the principal source of nitric acid in the future. While a categorical statement cannot be made, some of the major factors may at least be pointed out. The cost of converting nitrate of soda to concentrated nitric acid is just about equal to the cost of converting autoclave ammonia gas to concentrated nitric acid, interest and depreciation included in both cases. Ammonia gas, however, is a cheaper form of nitrogen than is nitrate of soda. It is cheaper by the amount of sulphuric acid required to fix the ammonia gas in the form of sulphate of ammonia. For nitrate of soda and sulphate of ammonia in the past have always sold at about the same price per pound of nitrogen. They will probably be sold on a competitive basis after the war, or if there is any difference the ammonium form will probably be the cheaper. The differential between ammonia gas and sulphate of ammonia, then, will make a difference of about 15 to 20 per cent in the cost of nitric acid in favor of ammonia oxidation. The fact that the nitrate of soda acid plants are being amortized during the war and are conveniently located for peace-time industrial uses, while new ammonia oxidation plants would have to be built at these same points in order to avoid transporting acid, is relatively not very important, because the interest and depreciation charge saved by amortization of the nitrate of soda acid plants is only about 4 per cent of the normal cost of the acid. The decisive factor will probably be simply the question whether the difference in cost of acid by the two processes is a sufficient incentive to overcome the inertia of human nature against changing existing practices.



Industrial Developments Relating to the Manufacture of Acetic Acid and Acetone

Tremendous Demand for Acetone for Explosives Led to Its Production from Molasses, Alcohol and from Calcium Carbide—Wood-Distillation Industry Faces Disaster Unless It Develops Scientifically

BY HAROLD HIBBERT
Director Research and Technical Division,
Ralph L. Fuller & Co., Inc.

PRIOR to 1914, more than half the entire world's supply of acetone and acetic acid was furnished through the medium of the wood-distillation industries of this country and Canada, the remainder being contributed principally by Austria-Hungary. When wood is heated in closed iron retorts in absence of air, it undergoes a decomposition whereby certain volatile constituents such as acetic acid, acetone and methyl acetate are driven off, the residue consisting of wood charcoal. In practice the crude acetic acid is neutralized by the addition of lime, which is then converted either into refined acetic acid or into acetone.

Acetic acid is used very extensively in the chemical industry for the manufacture of dyestuffs, pharmaceutical preparations, varnish for aëroplane wings and also as a condiment, while acetone finds its main outlet as a general solvent for varnish, lacquers, and in the manufacture of explosives such as cordite. This latter is used in immense quantities by the British and other Navies, and is made from a mixture of guncotton with nitroglycerine. In order to "compound" these materials, that is, to enable the gun cotton to become thoroughly incorporated with the nitroglycerine, it is necessary to employ a solvent which in this case happens to be acetone. Many other solvents have been tried out, but this has been found to give the best results. The British Admiralty early foresaw the necessity of obtaining large supplies of this material, and a few months after war broke out they were endeavoring to contract for an immediate supply of approximately 15,000 to 20,000 tons. This amount was considerably more than the entire world's annual production.

According to an article by Messrs. French & Withrow[1], the annual production of grey acetate of lime in this country in 1914 amounted to approximately 84,000 tons, since increased in 1917 to over 100,000 tons. This is equivalent to about 17,000 tons of acetone if all of it were employed for this purpose. As a matter of fact the greater part was used for the manufacture of acetic acid, the output of acetone being in the neighborhood of 7500 to 8000 tons annually. Faced with a sudden demand for an immediate increase in this commodity to some 20,000 tons, it was evident that new measures would have to be adopted for its manufacture, since it was altogether impossible to obtain this amount from the wood-distillation industry in the short time available. The assistance of the chemist and chemical engineer were therefore sought, and it is to the credit of the profession that they have provided an eminently satisfactory solution to meet this most urgent demand. The first line of attack was found in an attempt to increase the acetic acid production by the fermentation of various carbohydrate materials, as for example, molasses, through which alcohol and from this acetic acid in turn can be manufactured.

Working in coöperation with the British Government, the U. S. Industrial Chemical Co. has erected a large plant at Curtis Bay, and in spite of the many difficulties encountered both from a chemical, biological and engineering standpoint, they have so far succeeded that they are now manufacturing acetic acid, equivalent to approximately 70,000 pounds of 100 per cent strength per day, which is used for the manufacture of acetone and methyl acetate. This huge amount is all made from vinegar obtained from molasses alcohol by the quick fermentation process.

Some idea of the work involved in such an undertaking may be obtained when it is remarked that it was found necessary to construct special tank steamers for the conveyance of the molasses from Cuba to Baltimore. The generators comprise one series of 200 tanks, 25 ft. by 18 ft., and a second series of 960 of a size of 10 ft. by 18 ft. These are filled with beech shavings through the agency of which the oxidation of the alcohol to acetic acid is effected.

Anyone acquainted with the delicate nature of the changes taking place in such fermentations is well aware of the extraordinary care and oversight which it

[1] METALLURGICAL & CHEMICAL ENGINEERING. Vol. 13, p. 38.

is necessary to give to such operations to insure a commercial success, and the fact remains that the plant is now functioning day in and day out with continuous regularity.

To transport the dilute acetic acid liquors to the evaporators requires approximately 35 miles of wooden piping, and the evaporators installed for the concentration of the calcium acetate liquors, which are capable of handling some 200,000 gallons of liquor per day, require for this purpose a power consumption of 3000 horsepower.

While in all probability the manufacture of acetic acid and acetone by this process will not be profitable after the conclusion of peace, there is no doubt but that the company operating this plant will have first claim to be the largest manufacturers of alcohol in the world and the country should be proud of their supremacy in this respect.

Fernbach Process: Fermentation of Starch

Even with this additional amount of acetone and acetic acid available, it was evident that there would not be sufficient to meet the requirements of the British Government and their Allies, so that other processes came up for discussion. Among these was one relating to the "Fernbach" process. This consists in the fermentation of starch from corn or other grain by means of a special ferment which has the power of resolving the carbohydrate material into a mixture consisting essentially of butyl alcohol and acetone, the former largely predominating. The conditions of fermentation call for strict, careful chemical and biological control and those responsible for its development are to be congratulated on the successful results obtained. It is understood that the output of acetone at the plant in Toronto now amounts to 100 tons per month, while from the one in this country it approximates close to 250 tons. It would seem that the industrial disposal of the large tonnage of butyl alcohol formed would call for serious consideration, but it should offer no insuperable difficulties to the chemical profession.

Of the other processes calling for consideration, one of the most interesting was that relating to the manufacture of acetic acid from acetylene, regarding which a number of German patents had already been taken out some seven or eight years ago.

It has been known for a considerable period of time that when acetylene gas is passed through a solution of a mercury salt in dilute acid, it combines with the water, yielding a somewhat volatile liquid known as acetaldehyde, from which acetic acid in turn is readily obtained by the action of atmospheric oxygen. While those acquainted with the subject were aware of these developments, and with the fact that a plant had already been in operation in Germany for some two or three years prior to the outbreak of war, it nevertheless came as a great surprise to the majority of those interested that such was in reality the case.

The incident in fact merely furnishes another remarkable instance of the wonderful foresight of the German authorities in providing for their own requirements of such commodities, both from an industrial and especially from a military standpoint. To render themselves independent of this country in the matter of acetate of lime, research work was encouraged both in the universities and in the industry, and after laborious and painstaking investigations extending over many years, they were in a position to manufacture acetic acid and acetone from naturally occurring materials, namely, lime and coal, both available in large quantities in their own country.

Synthesis from Lime and Coal

Apart from a few experiments carried out by the writer while with the duPont Powder Company, nothing had been attempted in this country or Canada. However, the British Government was very fortunate in being able to obtain the hearty coöperation and assistance of the Shawinigan Light & Power Co., a Montreal corporation closely identified with the Canada Carbide Co. This concern on being approached by the British Government agreed to commence work immediately on the scientific aspect of the problem and was ably assisted in this connection by Prof. Ruttan of the McGill University, Montreal, Canada. They were fortunate in obtaining the services of Mr. Howard W. Matheson, formerly of the duPont Powder Co., and it is due to the chemical research investigations carried out by this chemist (ably supported as he was by the engineering skill of the vice-president of the company, Mr. Julian C. Smith) that they were successful within a period of one year not only in completing the experimental work (regarding which they were up to that time in complete ignorance), but also in erecting and starting the plant in actual operation.

The experimental work was started in December, 1915, and the plant was completed and commenced to operate in January, 1917, the total cost being somewhat over two million dollars. The output amounts to 56,000 pounds of 100 per cent acetic acid daily. When it is remembered that new and difficult questions of plant construction had to be faced, special type of construction relating to acid-proof materials and other important problems solved (in fact the creation of quite a new industry in this connection), and also that entirely new methods had to be devised in connection with each stage of the process, such as the electrolytic regeneration of the mercury etc., some idea may be obtained of the immense amount of labor involved. Not only was it necessary to build the chemical plant, but alongside of it a plant for the necessary calcium carbide had also to be erected and put into operation.

Second Plant Approaching Completion

When this country entered the war the question was discussed as to the advisability of erecting here a similar plant, but in view of the length of time it would take to develop the process and the saving which could be effected by coöperating with the Canadian Electro Products Co., an agreement was entered into for the duplication of the Canadian plant at an expenditure of over two million dollars. This second unit is now rapidly approaching completion and is expected to be in operation about December 1. The two plants will have an approximate production of around 55 tons per day of 100 per cent acetic acid.

The question as to what is to be done with this immense production after the war is one calling for serious consideration, as this is greater than the entire world's consumption. There would seem, however, to be no reason why the cost of manufacture should not

enable them to provide the industry with far cheaper acetic acid than was obtainable prior to 1914, and in this case the consumption will undoubtedly be largely increased in such industries as the manufacture of crude rubber, lacquers, dyestuffs etc. It should also be borne in mind that it is not at all improbable that extensive new industrial outlets will be found for acetaldehyde. This product, as far as its chemical reactivity is concerned may be said to bear the same importance to the so-called "aliphatic" chemical compounds as benzene does to the aromatic derivatives. From it by various chemical processes a whole series of valuable products practically constant. Recent researches have shown that it is possible to submit the wood tar to a process of distillation and by this means separate out certain of its constituents possessing marked germicidal action. This special property may then be intensified to a remarkable extent by certain simple chemical processes, thus increasing the value materially for purposes such as wood preserving, disinfectants, sheep dips etc.

There would also appear to be no doubt but that certain fractions of wood tar may be used to great advantage in the manufacture of varnish compositions and coatings, similar to the phenol formaldehyde con-densation products, while the pitch obtained as a residue when manufactured under certain well defined conditions is found to possess properties rendering it much more valuable than ordinary coal tar pitch for certain insulating purposes. Furthermore, there is a wide field in the application of certain constituents of these oils for use as protective coatings in the lining of acid vats, as extended experiments would seem to indicate that corrosion could best be guarded against in the handling of large quantities of acid liquors of various kinds by the employment of such materials.

THE CANADIAN ELECTRO PRODUCTS COMPANY

such as glycerine derivatives, solvents for nitro and acetyl cellulose, useful varnishes, camphor substitutes, synthetic rubber and numerous pharmaceutical products can be obtained, the development and exploitation of which will certainly follow on the conclusion of peace.

Wood Distillation Industry On the Defense

It is evident from the above that the wood-distillation industry after the war will have to fight for its existence, as the price of acetone will undoubtedly fall to a low level. In all probability the price of acetic acid made from carbide will be such that it will be impossible for them to compete. There is, however, a "life saver" in the situation in that the industry possesses in acetone, crude acetone oils, and wood tar, a very valuable source of, as yet, undiscovered revenue. If the wood distillation industry will only enlist the services of the industrial chemist, they will find that acetone can be readily and cheaply transformed into valuable solvents for varnishes and nitrocellulose lacquers. Moreover, it can be employed for the manufacture of synthetic rubber and glycerine substitutes, the latter possessing properties comparable to those of the ordinary glycerine of commerce. From the wood tar itself the well-known beechwood creosote can be made, the demand for which is increasing rapidly, while the production has remained

Germany and Switzerland Make Alcohol and Acetic Acid from Calcium Carbide

Nothing is clearer than the fact that unless the wood distillation industry awakens to the necessity of the employment of the trained research chemist and chemical engineer in the development of their business, the industry faces a very disastrous future. That this is the case is borne out by the fact that both in Germany and in Switzerland large electrical installations have been erected for the manufacture of alcohol and acetic acid from calcium carbide. The one in Switzerland is expected to use a minimum of 20,000 and a maximum of 30,000 hp. and produce annually 7500 tons of alcohol as a minimum and 10,000 as a maximum. It is stated

that this will make Switzerland an exporter instead of an importer of alcohol. The writer is informed by the American Consul in Berne, Switzerland, that the manufacture of acetic acid has already been commenced and that the manufacture of alcohol will be taken up later. In the Swiss Government reports on this subject, attention is drawn to the fact that Switzerland is dependent on this country and Austria-Hungary for its supply of acetate of lime. Government support for this undertaking was obtained with a view to making the country economically independent in this respect.

In Germany, according to the *Chemical Trade Journal*, the quantity of acetic acid produced in 608 vinegar factories amounted in 1913 to about 12,000 tons, while the quantity produced from calcium acetate and wood vinegar amounted to 23,000 tons of anhydrous acid. During the war several large factories for the production of acetate acid from carbide had been completed, while others are in course of construction with a view to the manufacture of artificial rubber. The factories if fully utilized are capable of producing 25,000 tons of acetic acid annually.

Much valuable research work has already been carried out under Government auspices at experimental stations such as the one at Madison, Wisconsin, and if full advantage were taken of these results as well as of the desirable coöperation of the industry, with such well equipped institutions as those at Syracuse, Cornell and Michigan, the future of the industry might be assured. As some of these institutions are supported by State funds, it is difficult to understand why their coöperation should not be sought and in fact insisted upon.

The article by Messrs. French & Withrow, referred to above, gives a very complete historical review and account of present day conditions in the wood distillation industry and contains much valuable food for thought in this connection.

New York City.

Dyeing With Coal Tar Dyestuffs

By *C. M. Whittaker*. Pages, 214. Price $1.80. London: Baillière, Tindall & Co.

The author has been in charge of the experimental dyehouse of one of the larger British dye manufacturers for a number of years and while he has not undertaken to teach the novice to be a successful dye artist, he has made a great deal of information on this subject more interesting as well as easily capable of assimilation. The point of view throughout the book has been based on the commercial requirements. Basic dyestuffs are described with reasonable detail and the scope of use of this class of colors indicated. Acid dyes, alizarine, direct cotton, azo, resorcine, sulphur, vat, oxidation and compound textile dyes are all treated in a very readable manner.

Mr. Whittaker says, "The ever present demand for cheapness must always be recognized, however much one may regret it. Big production in modern industry takes the form of a cheap, not a dear, article. A constant endeavor is being made by cloth designers to make as good an imitation as possible of a costly fabric of a cheaper material. This explains why one now meets so many fabrics which contain wool and cotton to imitate at a low price the more expensive all-wool article. To give an example, a cotton warp is woven with a woolen weft in a serge pattern to imitate an all-woolen serge. These fabrics have to be dyed, and reflect themselves in the increasing demands for union dyestuffs, which has been one of the most striking features in the modern coal-tar dye industry." The subject of dyehouse machinery is not gone into, the book being intended to supplement the average chemist's training in preparation for textile work.

The Manufacture of Enamel-Lined Apparatus*

By Emerson P. Poste

THE field of enameling includes enamels applied to steel and those applied to cast iron. Under the former heading we have cooking ware, signs and other miscellaneous pieces and apparatus used for large-scale food preparation and the chemical industries. Cast iron includes sanitary ware, various stove parts and other decorative pieces and apparatus for chemical and pharmaceutical purposes.

In the preparation of an enamel, various raw materials such as feldspar, borax, soda, cryolite and fluorspar are used for the basis of the mixture. Various colors are produced by the use of cobalt, manganese and other coloring oxides some of which are added in the original mix and others in the mill.

These raw materials are thoroughly mixed and placed in a hot smelting furnace, where the materials are melted together and drawn out into a tank of water and granulated. The product is called frit. This frit is ground either wet in a pebble mill with water, clay and other mill additions, or ground in a dry mill for application by the dry process.

The steel ware to which the enamel is to be applied is manufactured either by pressing, drawing, spinning or welding. Whatever the manner of preparation or the nature of the piece, the surface to which the enamel is to be applied must be clean. This is accomplished either by pickling or sandblasting, depending upon the size.

The application of the enamel by the dry process on cast iron involves the spraying of a preliminary ground coat which when dry is placed in the furnace and withdrawn at a red heat. The dry powder is dusted on, the piece put back in the furnace, the enamel melted down, the piece withdrawn, more enamel added, and so on until a sufficient coat is built up.

In the wet process the enamel after drawing from the wet mill is treated with electrolytes to make a proper consistency for spraying or other method of application. The piece is then either dipped in the enamel, the enamel poured over or sprayed on, depending upon the size and shape of the piece.

In the burning of the ware two general types of furnaces are used, direct-fire and muffle. The latter is more general in connection with cooking ware and smaller pieces while the former is used on some of the larger pieces used for chemical work and large-scale food preparation. In either case the burning of the enamel consists in melting down the individual particles and fusing them together in the form of a continuous coating.

Apparatus made in accordance with the above general outline ranges from 50-gallon to 4500-gallon units in steel and from 2-gallon to 300-gallon in cast iron. The former line finds application primarily in the dairy, canning and other food industries. Units are also used as storage, evaporating and reaction tanks for chemical processes of a neutral or slightly acid nature. The cast iron equipment is used as above and in addition for the most severe chemical conditions involving mineral acids.

*An abstract of a paper read at the Fourth National Exposition of Chemical Industries, New York, Sept. 24, 1918. The paper was illustrated by lantern slides, and there was an exhibit of products.

American Dyes from a Manufacturing Standpoint

Domestic Dyestuff Industry Becoming an Accomplished Fact—Analysis of Our Needs and Figures of Actual Production—Permanence of Industry Depends on Tariff

By W. H. WATKINS

THE developments growing out of the Great War made it evident by the Spring of 1915 that we should be obliged to look to ourselves for our accustomed supply of dyestuffs. There was no question of going without them. The great textile, leather, paper and other industries were in great part dependent upon dyes for the production of merchantable goods. Natural dyestuffs could in part fill the gap caused by the elimination of Germany as a source of supply of coal-tar dyes; but natural dyestuffs could not be obtained in sufficient quantity, nor were they in many cases acceptable substitutes either by reason of lack of fastness or variety of hue, or because of difficulty of application. If the textile, paper, leather and other industries giving employment to perhaps two million people and producing goods of a value of perhaps four billion dollars were to continue doing "business as usual" an American coal-tar dyestuff industry had to be created.

To the chemist the outlook was most enticing. What could be finer than the opportunity for working out methods for the manufacture of hundreds, perhaps thousands of beautiful compounds, beautiful to the chemical sense as well as to the eye. The manufacturer, chemist or not, who contemplated engaging in this industry however, could not consider alone the alluring chemical side of the problem. American chemists had always found this coal-tar chemical industry a most interesting industry to look at and talk about, but also had generally concluded that it was a very safe industry to let alone. The few chemists or manufacturers who did take hold of it were generally found after a few months, or a year or two, vigorously blowing their fingers.

Figures and Statistics Not Alluring

So it was well for the manufacturer who felt the lure of coal-tar dyes or chemicals to look over the chemical charts before embarking on the deep waters of their manufacture. The most recent charts available in the spring of 1915 were not particularly encouraging. An eminent ex-president of the American Chemical Society had demonstrated to the United States Chamber of Commerce that it was distinctly a "one-nation" business. He made a good case of it too. He showed that the import value of the coal-tar chemicals used in the United States was less than the value of the confectionery sold over the 5-&-10-cent counters.

Our most eminent student of chemical statistics in a most comprehensive study pointed out the complexity of the industry and its difficulties, and concluded that we could have the industry if we were willing to pay the price. The question the manufacturer had to ask himself was: Who is to pay the price?

The would-be manufacturer also found that the duty-paid value of the coal-tar dyes and chemicals, assuming the adoption of the highest duties to be hoped for, would not be over $15,000,000 per year on the basis of pre-war values. He further found according to authoritative statements that 922 different dyes were necessary, these being dependent upon about 300 intermediates made from ten coal-tar crudes by the use of vast quantities of acids, alkalies and other chemicals.

The business had a pot-walloping look at the best, and the American genius for production has not been of the pot-walloping order. However, the statement in regard to 922 dyes referred to above was based on the number of dyes listed in the 1914 edition of the *Schultz-Tabellen* and not on an analysis of the American dyestuff problem as it existed.

Formulating the Real Problem

The problem for solution by the American dyestuff manufacturer might have been stated somewhat as follows: To provide such a line of coal-tar dyes as in variety of hue, method of application, and fastness, will meet the needs of the textile and other manufacturers in these respects, having in mind the requirements of the ultimate consumer for service on the one hand, and the exigencies of manufacture on the other.

An analysis of the problem so stated made it seem possible to meet the legitimate demands of the situation with about 300 dyes derived from about 100 intermediates. Further analysis seemed to indicate that it might be possible to put the main part of the business on something better than a pot-walloping basis. If the industry is to be carried on by a large number of small concerns however, each endeavoring to bring out a complete line, it will be a business of small units and high labor costs.

In the following analysis the estimated needs of the whole country for the volume and kind of manufacture carried on before the war, has been taken as the basis.

The war conditions of today, however, have "busted the whole plan of salvation." But the war must be over some day, and the manufacturer who would survive must look forward to that day as well as take care of the emergency needs of the present.

Analysis of United States' Dye Needs

An annual American consumption of 50,000,000 lb. of coal-tar dyestuff has been assumed, and the information disclosed by the "Norton census" has been applied to the original estimates.

The necessary dyes were placed in five classes according to the quantities needed annually.

First Class.—Consumption in excess of 1,000,000 lb.

per year. Eight dyes fell in this class with a total of 25,000,000 lb. or one-half of the whole.

Second Class.—Consumption less than 1,000,000 lb. but 250,000 lb. or more. Twenty-seven dyes fell in this class with a total of 10,500,000 lb.

Third Class.—Consumption from 100,000 to 250,000 lb. Fifty-one dyes fell in this class with a total of 7,100,000 lb.

Fourth Class.—Consumption from 50,000 to 100,000 lb. Forty-seven dyes fell in this class with a total of 3,200,000 lb.

Fifth Class.—Consumption less than 50,000 lb. Approximately one hundred and sixty dyes would fall in this class with a total of 4,200,000 lb. or about 25,000 lb. each on an average.

So unless production of the same dyes was undertaken by too many independent concerns, a good part of the dyestuff manufacture could be put on a respectable production basis; that is, advantage could be taken of labor-saving devices and materials could be routed with some degree of order and system.

Chemical Problems in Producing Intermediates

But there is another and more complex feature of the problem. To make these dyes the intermediates must first be made. We may estimate that our program will call for 100,000 lb. of an ortho isomer and find that in producing that amount of ortho we also get 200,000 lb. of the paraisomer for which we have very little outlet. The ultimate success of the American dyestuff industry will depend largely upon the correct solution of just such problems. The solution of such problems will inevitably compel the manufacturer who would limit his business to a few individual dyes to increase his line, and increase will bring additional problems. But the manufacturer who piles up his para in order to sell ortho will certainly burn his fingers if he sticks to it.

Assuming that the 300-dye program is reasonably correct, the present status of the industry is encouraging. The estimate of the present status as here given is not based on the manufacture of any one concern but on the entire production of the country as far as it can be gaged by the products now on the market. The figures given below should not be taken as absolute. They are, however, approximately correct.

Our Present Production of Dyes

Of the eight dyes placed in class 1, seven are being made now, in several cases in amounts considerably in excess of the original estimates.

Of the twenty-seven dyes in class 2, nineteen are now on the market and plants for the remaining eight are already in process of construction or projected.

Of the fifty-one dyes in class 3, twenty-six are now on the market and plants for nineteen of these remaining are being constructed or projected.

Of the forty-seven dyes in class 4, seventeen are now on the market and of the remaining thirty, plants are either under construction or projected for fifteen.

Of the large number of dyes in class 5, about sixty are on the market and sixteen are in prospect.

Not all the dyes mentioned as on the market are there in sufficient amounts, but if they can be made, it is only a question of increased plant to produce them in sufficiency. The main problem as far as they are concerned has been solved, provided some inconvenient, not to be disposed of, isomer does not enter into consideration.

Development is Proceeding Logically

It is evident that the development of the American industry has proceeded on correct lines from the standpoint of the interest of all concerned. Those dyes required in largest amounts have actually been made first, and new projects have been undertaken roughly in the order of their importance as measured by the amount consumed.

This perfectly correct policy, however, has made it necessary to delay the production of some dyes, the importance of which is not measured entirely by the amount consumed. These dyes are those requiring comparatively small amounts of special intermediates. Obviously, such dyes require quite as much chemical and engineering investigation as those needed in larger quantities. It has certainly been the wisest course to apply the available chemical and engineering skill in such a way as to have it result in the production of the greatest possible amount of the commodities dependent upon dyes. The problems already solved also have their application in the solution of those as yet unsolved. There are few types of reaction employed in manufacturing dyes with which American chemists and engineers are not now familiar. The remaining problems, therefore, present fewer difficulties than the earlier ones. Then, too, many of the dyes needed in comparatively small amounts will appear simultaneously with dyes of greater importance, as the intermediate necessary for an important dye is frequently the one intermediate lacking for the production of a number of less importance.

With a few gaps here and there, the needs for acid, basic, chrome, and direct dyes are fairly well met with the dyes now available. With those in prospect, practically all needs will be satisfied for these classes.

Quality of Domestic Product Equal to Imported

Well-established American dyes made today by recognized manufacturers are equal in quality to the same dyes as formerly made in Germany. It is true that a considerable number of dyes formerly made in Germany are not as yet made in America. The lack of these dyes is partly responsible for the lack of fastness to light and wear being sometimes observable in textile fabrics. As a rule, however, such lack of fastness is due to faulty application, or to the wrong choice of available dyes for the job in hand.

Alizarin has appeared and its derivatives will follow. Vat dyes will not be long delayed. With good fortune, including a reasonable supply of chemists, labor, acids, toluol and phenol, the next year should see the American dyestuff industry a complete, accomplished fact.

Important as the establishment of this industry is for America, it is much more important that the war be won. Many of the essentials for making dyes are also essential for maintaining the military establishment, and the military establishment comes first. As between chrysophenine and TNT there is but one choice. So it may be safer to say that, subject to winning the

war, the next year will see the American dyestuff industry an accomplished fact.

The permanence of the industry is another matter. With Germany able after the war to lay goods on the dock in New York at pre-war prices, its permanence may be doubtful under existing tariffs. America has been shown that she can have a dyestuff industry at a price. What this price in the way of tariff protection may be no one can now estimate. But America knows the price she has paid for the lack of a dyestuff industry and will undoubtedly be willing to pay the price to keep the one she has gained.

American dyestuff manufacturers appear to be proceeding as though assured of a permanent industry.

Buffalo, N. Y.

Ordnance Department Establishes Industrial Education Section

The Army Ordnance Department announces the establishment of an Industrial Education Section for the purpose of increasing productive efforts in the plants engaged in the manufacture of ordnance material.

The activities of the section are under the direction of Major F. L. Thompson and Captain C. R. Dickinson, and will be aimed to carry home to the individual worker, be he employer or employee, the fact that his own personal efforts will have a direct bearing on the winning of the war. The agencies employed include posters, booklets, pay-envelop stuffers, newspaper publicity, display advertising, motion pictures, public speakers and exhibits.

Posters carrying a message of encouragement to the worker tending to inspire greater individual effort are sent out to the manufacturing plants at regular intervals. Some of these, notably "Not Just Hats Off to the Flag, But Sleeves Up for It," have been remarkably successful, according to the more than 900 letters received by the Ordnance Department regarding it.

Booklets and pay-envelop stuffers are used to convey inspirational messages to the home and thereby tend to increase the morale of the community. An edition of 2,000,000 of one of the smallest posters ever issued, bearing a message to the individual, was distributed in a number of cities on Labor Day by means of aëroplanes dropping them over the course of the parade.

In the neighborhood of 1000 plants working on war materials now have motion-picture projection rooms and have been furnished films with a distinct appeal to the worker, such as the Government films, "Pershing's Crusaders" and "America's Answer." These films have proved very successful in definitely linking up the work of the man at the lathe with the boys at the front.

In the same manner, speakers, many of them foreign soldiers returned from the front, have been furnished to meetings of plant employees, and have given their hearers first hand information as to how necessary it is to speed up production.

Advertising and publicity in the newspapers by local communities has been advocated and copy furnished from Washington whenever desired.

In some instances, it has been found desirable to encourage public exhibitions of locally made material used by the army as a means of arousing local pride and stimulating interest in war work. Several exhibitions of this kind illustrating the use to which the material was put in actual service, have been the direct means of increasing the pride of the citizens in their town and consequently in their own work.

In order to centralize the work and afford increased service to the various war working plants, it has been decided to appoint a representative of the Industrial Education Section in each of the eleven District Ordnance Offices as follows:

Bridgeport, Conn., Ralph B. Boas, formerly instructor at Brown and Harvard universities and a writer and lecturer of note; Boston, D. D. Cassidy, architect; New York, Major J. C. Mitchell, U.S.A., formerly president Etheride & Company, advertising agency; Philadelphia, J. W. Watson, president of the American Bronze Company; Rochester, N. Y., William H. Complice, Rochester Chamber of Commerce; Pittsburgh, Charles Yon, Pittsburgh & Lake Erie Railroad; Detroit, W. G. Pipp, editor Detroit *News;* Cincinnati, Carl H. Jacobs, Jr., vice-mayor and former assistant city solicitor; Chicago, D. L. Benjamin, of Gray & Benjamin, advertising agents; St. Louis, Homer Hall, assistant district attorney. The representative for the Cleveland district has not as yet been appointed.

These men are to be responsible in their respective sections for the activities of the Industrial Education Section which will act as a service agency at the disposal of the various District Ordnance Chiefs to assist them in increasing production effort.

Chemically Treated Fabrics for Powder Bags

Chemically treated cotton cloth, as a substitute for silk, is being tested out by the Ordnance Department. If found practicable for ordnance uses, the discovery will effect the double result of meeting a serious shortage in silk, and of bringing about a money saving in the ordnance program estimated at between $25,000,000 and $35,000,000.

Preliminary tests already made at the Aberdeen Proving Grounds have encouraged the department to proceed further with its experiments; and for this purpose an order for 5,000 yards of the new material has been placed with the concern responsible for developing the process of treating the cotton cloth.

At present millions of yards of silk are required in making the bags which contain the large powder charges used in the firing of heavy artillery. These bags are inserted in the gun immediately behind the projectile, and the firing of them gives the propelling force that hurls the projectile at the target. This propelling charge is, of course, entirely distinct from the charge within the projectile that explodes the missle after it reaches the target.

Heretofore silk has been depended upon for these bags for the reason that no other cloth material has been found that would meet the peculiar conditions required. It is essential that not a particle of the bag container shall remain after the gun is fired. Otherwise a smouldering piece of the fabric might cause a premature explosion when a new charge was inserted.

Large-Scale Sulphuric Acid Manufacture

A Description of the Plant of the Tennessee Copper Company Where the Special Features in Both Construction and Chamber Control Are as Noteworthy As the Fact that This Plant Is the Largest in the World

BY ANDREW M. FAIRLIE
Consulting Chemical Engineer, Copperhill, Tenn.

THE large plant for the manufacture of sulphuric acid may consist of either a large number of small or average units, all located at a single site and operated under one management, or a few units of unusually large proportions. In the former case the equipment and methods of operation need present nothing of extraordinary interest. The chambers, towers, fans, blow-cases, etc., may all be of a type and size with which the average acid-maker is familiar, and the operation of a number of small plants presents no difficulties or unusual situations which are not encountered in the operation of a single small plant.

An example of a large plant composed of a large number of units will be the new sulphuric acid plant now being erected for the U. S. Government at Brunswick, Ga. This plant will consist of eight units, each of 100 tons capacity. A 100-ton unit was formerly considered a large plant, but now that there are in this country so many units two or three times that size, the 100-ton unit has almost shrunk to average size by comparison. Another large plant, consisting of a large number of small units, is the plant of the United States Steel Corporation at Donora, Pa. This plant, comprising six units, each of 75 tons capacity, has a total capacity of 450 tons daily. Such plants as these, being a multiplicity of small or average units assembled or constructed at one site, contain presumably only the usual equipment, common to plants of average size anywhere.

THE PLANT OF THE TENNESSEE COPPER COMPANY

It is the large plant composed of one or more extra large units that presents features of more than ordinary interest. The plant of the Tennessee Copper Company, located on the Ocoee River in the southeastern corner of Tennessee, is recognized as being not only the largest sulphuric acid plant in the world, but as being composed of the largest units in the world. The Tennessee Copper Company enjoys the distinction of being the original pioneer in the manufacture of sulphuric acid by the chamber process from the waste fumes of copper blast-furnaces. It is indeed fortunate that years ago the difficulties at first encountered were overcome, and that long before 1914 the enterprise had been put on a permanent and reliable basis, so that now, when acid is so much needed, and when sulphur and pyrites are so scarce, this large supply of sulphuric acid, made as a by-product, may be forthcoming to meet the needs of the Government.

The sulphuric acid plant of the Tennessee Copper Company comprises Plant No. 1 and Plant No. 2. Plant No. 1 is composed of two units, each of an average daily capacity of 300 tons (60 degrees Baumé). These two units were formerly a single unit of 600 tons daily capacity, but in 1917 this large unit was subdivided into two approximately equal units. No. 2 Plant consists of one unit only, of a capacity of approximately 300 tons.

The five prominent features of a sulphuric acid plant are the burners, for generating the sulphur dioxide gas; the niter pots or retorts, for generating the oxides of nitrogen from nitrate of soda; the Glover tower, for denitrating the nitrous vitriol, concentrating the chamber acid, furnishing a part of the steam for the chambers, and cooling the hot burner gases; the lead chambers, the oxidizing, hydrating, and condensing apparatus; and the Gay-Lussac towers, for recovering the oxides of nitrogen in the form of nitrous vitriol. Less prominent, but quite as important features, are the gas fans, the acid pumps or blow-cases, the acid coolers, the hydration supply system, and the tanks.

The plant of the Tennessee Copper Company has no burners. The sulphur dioxide gas is generated in the copper blast-furnaces, and is conducted through a large brick flue to the Glover towers. This flue, the cross-sectional area of which is 200 square feet, is over 200 feet long to No. 1 Plant, and more than 500 feet long to No. 2 Plant. The percentage of sulphur dioxide in the gases ranges usually between 3 per cent and 6 per cent and the temperature of the gases entering the Glover tower may range between 600 and 900 deg. Fahr.

EXTERNAL NITER RETORTS AND AUTOMATIC CHARGERS

The niter retort system now in use at these plants is unique. The common method of "potting" niter is to have the pots inside the hot-gas flue leading to the Glover tower, and to rely on the heat of the burner or furnace gases to distil the niter fumes from the pots. This method was formerly used at the No. 1 Plant of the Tennessee Copper Company. But it was found that some portion of the charge would overflow the pots, and from time to time a pot would develop a leak, permitting some of the charge to run out of the pot; and though due provision was made for tapping out such molten overflow or leakage from the pans underneath the pots, nevertheless, some of the overflow and leakage would inevitably solidify inside the hot-gas flue. Crusts of solid niter cake inside the flue would grow in size, until in course of time a solid mass would be formed in the flue, thus partially blocking the flue for the passage of gas and preventing the gas from getting free access to the pots in order to heat them, so that under these conditions it was neces-

sary to shut down the entire plant, once or twice a year, for ten days or more, in order to clean the niter cake out of these flues.

For this reason the pots inside the flue were abandoned, and a system of retorts outside the flue and fired with coal was installed. The retort adopted is the standard retort used in nitric acid manufacture, and is shown in Fig. 1.

This retort is ten feet high and eight feet in diameter, and is large enough to cook a charge of 8000 pounds of nitrate of soda without overflowing. The retorts are built in groups of three retorts to a chamber unit, but usually two retorts are sufficient to operate a unit, the third being held in reserve as a spare. Fig. 2 shows one of these groups of retorts.

Each retort is supplied with niter by an automatic feed device, motor-driven, and geared to a lead wheel provided with lead cups burned to its circumference, revolving in a small tank of acid in unison with the

FIG. 1.—RETORT

revolutions of the worm which feeds the niter, so that automatically, as the niter is fed, the correct proportion of sulphuric acid is poured into the pot. Fig. 3 shows a close-up view of this mechanical feed for niter and acid.

The Glover towers of these plants are octagonal in shape, are 55 feet high and 30 feet in diameter from lead to lead. The lead curtains are lined with chemical brick laid in acid-proof cement. The thickness of the brick walls at the bottom of the tower is 22½ in. The towers are packed with acid-proof tile. The amount of acid pumped over the three towers, combined, including nitrous vitriol and chamber acid, is about 3600 tons, or 72 tank car loads per day. The chamber acid is concentrated to a strength of about 60 deg. Bé. The temperature of the acid discharged from the Glover tower is about 280 to 300 deg. F., and of the gas escaping from the tower into the chambers, about 200.

Six Million Cubic Feet of Chambers

The lead chambers at No. 1 Plant are 34 in number, and are of unusual height. There are eleven chambers, 50 x 50 x 67 feet high six chambers 50 x 50 x 72 feet high, four chambers 18 x 50 x 77 feet high, four chambers 10 x 50 x 67 feet high, and five chambers 67 feet high, but of varying lengths and breadths. The total chamber space at No. 1 Plant is 3,944,800 cu.ft. At No. 2 Plant there are only four chambers altogether, but these are probably the largest acid chambers ever built. The dimensions are 59 ft. wide x 236 ft. long x 40 ft. high, each chamber enclosing 556,960 cu.ft. of space.

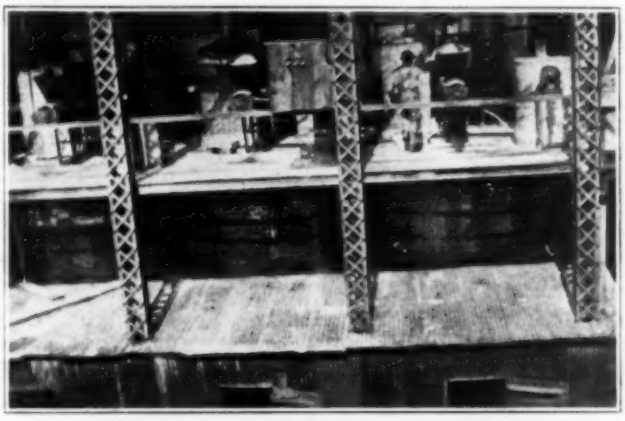


FIG. 2.—NITER RETORT GROUP

The total chamber space at No. 2 Plant is 2,227,840 cu.ft. For both plants combined the total chamber space is 6,172,640 cu.ft. At No. 1 Plant the chambers are connected by straight flues, these flues being alternately at the tops and bottoms of adjoining chambers. As most of the alleys between the chambers are only seven feet wide, most of the chamber connections are quite short. An exception to this rule is the flue con-

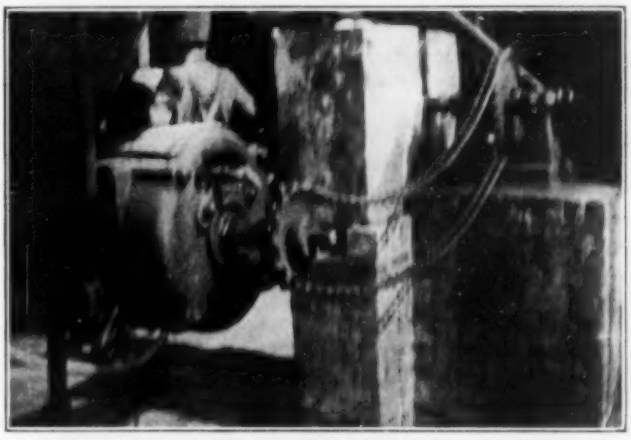


FIG. 3.—AUTOMATIC FEED

necting No. 6-B chamber with No. 7-B. These two chambers are so located that they are about 225 feet apart, and it was necessary to build a special flue to connect them. This flue, octagonal in shape and having a cross-sectional area of 100 sq.ft. and a diameter of 11 ft., has become quite famous throughout the country as the flue "big enough to drive an automobile through"—which is literally true of it. This flue has been referred to sometimes as the "Zeppelin Flue," from its resemblance to a Zeppelin airship. It is shown in part in Fig. 4, and its size looms large in comparison with the man standing beside it. The cross-sectional area of the other chamber connections at No. 1 Plant is approximately 100 sq.ft.

At No. 2 Plant the connections between the chambers are usually only seven feet long. But here each pair of adjoining chambers is connected by two connections (instead of by one only as at No. 1 Plant), each with a cross-sectional area of 25 sq.ft. There is one long round flue, connecting the east side of No. 2 chamber with the west side of No. 3 by means of a return bend, whose diameter is 5 ft. 9 in.

Special System of Gas Mixture Control

The chambers at both plants are provided with the usual equipment of thermometers and hydrometers for regulating the chamber temperatures and the strength of the "drips." While these instruments are used as aids in the control of the process, the quantity of niter admitted to the plant is regulated in accordance with the indications of the analytical method of chamber process control, originated by the author at this plant in 1910, and in continuous use there ever since.[1] This method of control has proved very useful at these

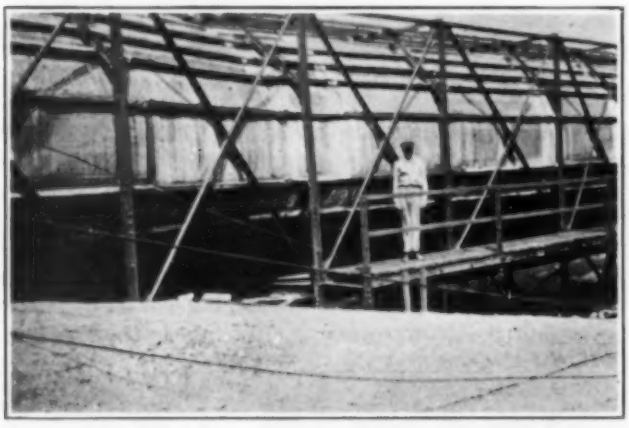


FIG. 4.—SULPHUR DIOXIDE FLUE

plants, on account of the fluctuating character of the furnace gases, and its use has resulted in a large saving of nitrate of soda. The method was described in detail in the *Transactions* of the American Institute of Chemical Engineers, Vol. IX (1916), pp. 319-332, and for a complete understanding of the method, the reader should refer to this publication.

Briefly, the method consists in the establishment, as the result of experiment, of a series of desirable ratios between the percentage of sulphur dioxide in the gas entering the Glover tower and the percentage of sulphur dioxide in the gas mixture at any fixed point in the front chamber, covering the entire range of sulphur dioxide percentages which are likely to be encountered; and in maintaining thereafter such desirable ratios constantly, by introducing the right amount of nitrate of soda for the purpose. Once the series of desirable ratios is established, they are arranged in tabular form, and the table is framed and posted before the eye of the chamberman. This man tests frequently the percentage of sulphur dioxide in the gas at the point in the front chamber chosen for the test, and by comparison with the percentage of sulphur dioxide in the gas entering the Glover tower at about the same time, knows by reference to the established table whether the amount of niter going into the system is too little, too much, or just right. If any error in the amount is made, the error is detected in the front chamber, and the mistake is corrected before any serious damage is done. By this means it is possible to maintain uniformly the desirable percentage of sulphur dioxide in the gases escaping from the last chamber (from 0.06 to 0.12 per cent under normal conditions) thus securing the most efficient recovery of nitrous oxides in the Gay-Lussac tower.

[1]See U. S. Patents, Nos. 1,205,723 and 1,205,724.

Two Types of Gay-Lussac Towers

The Gay-Lussac towers at No. 1 Plant are eleven in number, and are of various shapes and sizes. Three of these towers, octagonal in shape, are 62 ft. 4 in. high and 35 ft. 7 in. in diameter from lead to lead; four smaller towers, also octagonal, are 70 ft. high and 19 ft. in diameter from lead to lead; and four square towers are 47 ft. high and 23 ft. square. These towers have lead curtains, lined with acid-proof brick walls laid dry; some are packed with quartz, and others with acid-proof brick or tile.

At No. 2 Plant the Gay-Lussac tower is of peculiar construction. There is only one tower, and it is built of acid-proof brick laid in acid-proof cement, without lead curtains. The tower is divided into 56 cells or compartments, the partitions being of acid-proof brick. The arrangement of the compartments is eight parallel rows of cells, seven cells to the row. The gases traverse the eight rows in parallel, and pass up one cell and down the next (and so on) of each row, until, arriving at the top of the seventh cell, they are drawn through the back fan and are then expelled through the exit stack into the atmosphere. The cells are each eight feet square inside, with a total height of 40 ft. They are packed with acid proof tile laid on edge. The distribution of the acid over the tops of the cells is effected by means of a single acid feed-pipe to each cell, terminating in a sprayer provided with a splash-plate inside the tower, by means of which the acid is quite evenly sprinkled over the entire top of the tower packing. The lead pan at the bottom of the tower is divided into three compartments by means of brick partitions, whereby it is possible to keep separate nitrous acid of three different grades of nitrosity, as discharged from the tower. The effect, then, of this peculiar tower, so far as the feed and discharge of the acid is concerned, is that of three towers arranged in series—the first (the one nearest to the chambers) consisting of eight rows of three cells to the row, over which the acid discharged from the middle group is fed, and from which the nitrous vitriol which is fed to the Glover top is obtained; the second, consisting of a continuation of the same eight rows, but with only two cells to the row, on which the acid discharge from the last group of cells is fed; and the third, consisting again of a continuation of the same eight rows of cells, two cells to the row, over which cooled acid discharged from the Glover tower is sprayed. This Gay-Lussac tower, or cluster of little towers, is of interest because of its peculiarity. After two years of operation, no particular advantage in this type of tower has been observed, but serious obstacles or disadvantages have been encountered, chief among which is the difficulty of washing the cells when they become clogged with acid sediment.

An unusual quantity of sediment is deposited from the acid made at these plants. This is because of the flue-dust blown out of the furnaces by the blast. At the ordinary plant, air is admitted to the burners or furnaces in a natural way, as induced by the draft of the acid plant. But in the case of the copper blast-furnaces air is blown into the tuyeres at the bottom

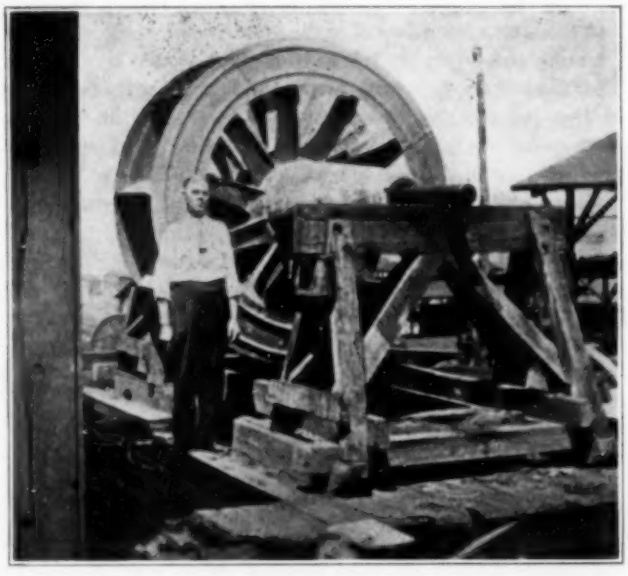


FIG. 5.—IMPELLER FOR SO_2 GAS

of the furnaces under a pressure of about four pounds per square inch. This blast inevitably forces large quantities of fine particles of the furnace charge out of the furnace, and although brick chambers have been provided for settling the dust before the gas reaches the Glover tower, a substantial quantity of the finer dust enters the Glover tower with the gas, and encountering there the streams of acid flowing down the tower, is washed out, to form a stiff paste which collects at the bottom of the tanks. But not all of the solid impurities are eliminated from the gases, even in the Glover tower. Zinc fume, in particles so fine as to escape being washed out by the generous streams of acid pouring down the Glover tower, finds its way through the tower packing into the lead flues leading to the gas fans, and in the fans most of it is deposited as impure zinc sulphate, partly in the bottom of the fan casing, and partly on the vanes or blades of the impeller. A part of the zinc fume even goes on past the fan, to be deposited as zinc sulphate in the bottom of the chambers.

Gases Propelled With Lead-Antimony Blowers

As would be expected, the furnace blast produces a pressure on the hot-gas flues. This pressure is not sufficient, however, to force the gas through the chambers at the desired rate of speed; and moreover if reliance were placed exclusively on the furnace blast as motive power for the gases, the pressure on the hot-gas flues and on the furnace tops would be so great that not only would a large proportion of the gases be lost, but conditions on the furnace charge floor would be intolerable for the smelter workmen. Gas fans, therefore, have been installed for the purpose of drawing up the gases from the furnaces, and forcing them on through the chambers. Gas fans are a common, and, indeed, a necessary adjunct, of any modern acid plant, but are usually much smaller in size than the fans about to be described.

The impellers of these fans, made of antimonial lead, are eight feet in diameter, and the vanes, of which there are ten to each impeller, are four feet wide. The vanes are attached to the hub by means of twenty cast arms or spokes. Keyed to the cast lead hub there is an inner hub of cast iron, and to this the steel shaft is keyed. The complete impeller, including shaft, cast-iron hub, lead hub, arms, vanes and shields, weighs approximately six tons. These impellers are made at the shops of the Tennessee Copper Company, and several have been made there for some of the large plants of the far West, which use this type of gas fan. Fig. 5 shows this impeller, and Fig. 6 shows the external appearance of one of these gas fans, the impeller being concealed by the housing.

Blow-Cases Superior to Other Elevating Devices

At No. 1 Plant seven of these fans are in operation, four at the front end of the chamber system and three at the back. At No. 2 Plant there are two front fans and two back fans.

Various means of pumping acid have been tried at these plants—blow-cases, electrically-driven centrifugal pumps (both horizontal and vertical types), electrically-

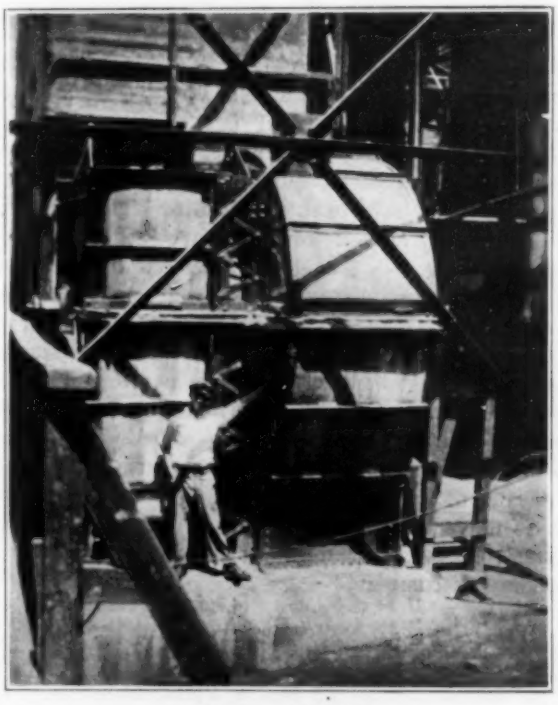


FIG. 6.—GAS FAN

operated skip-hoists, and air-lifts, and while some of the newer devices for elevating acid have shown lower costs than blow-cases as regards power consumption, from the standpoint of efficiency, operating labor required per unit of acid pumped, cost for repairs, total operating cost, and freedom from interruptions, no elevating device tried has equalled the compressed-air blow-case. For this reason all acid pumping done at these plants, with the exception of the elevating of the chamber acid (for which the air-lift is used) is now

done by means of blow-cases. The blow-case usually met with at an acid plant is made of cast iron; but at these plants blow-cases made of riveted wrought iron plates were adopted ten years ago, and have proved so successful that there has never been a cast-iron "egg" for pumping sulphuric acid on the place. The feed lines to these blow-cases are 6-inch iron pipes, and the discharge lines are 4-inch iron pipes.

Necessarily, for the purpose of cooling the enormous quantity of acid discharged from the Glover towers of these plants, coolers of special design had to be constructed. At No. 1 Plant there are six coolers, rectangular in shape, the inside dimensions being 17 ft. long x 8 ft. 6 in. wide x 4 ft. 8 in. deep. The frame-work is of steel channels placed edgewise to the cooler, supporting a shell of 15-lb. lead, which is lined with 9 inches of acid-proof brick laid in acid-proof cement. Each cooler contains 71 coils of 1½-in. lead pipe, the coils being 12 inches in diameter. In the six coolers combined there are over four miles of 1½-in. lead pipe. Three of these coolers are built in terrace form so that the acid flows by gravity from one cooler to the next, and this is found to be an advantageous arrangement. At No. 2 Plant there are three coolers of the same type and size as those at No. 1. By means of these coolers the Glover tower acid is cooled from about 280-300 deg. to 100-110 deg. F.

Atomized Water Used in Chambers in Place of Steam

The hydration of the acid in the chambers of these plants was formerly accomplished by means of steam. Water sprayers had seemed out of the question on account of the limited capacity of the sprayers on the market. An enromous number of these low-capacity sprayers would have been required to supply the quantity of water needed for the large-scale operations here under way. A special atomizer has been developed at this plant, however, capable of atomizing as much as fifty gallons of water per hour, and with this device water-spray has been substituted for steam in most of the chambers, resulting in a considerable saving in coal, lower temperatures in the chambers, larger chamber capacity, and apparently some saving in niter consumption. As the water for the sprayer is pumped by means of centrifugal pumps operated by electrical motors driven by water-generated current, the saving of the coal formerly used to generate steam for the chambers is complete.

Wrought-Iron Blow-Cases and Tanks Being Installed

The factory tanks at No. 1 Plant have been, until lately, of the usual wooden construction, lead-lined. When No. 2 Plant was built, in 1916, on account of the acid-resisting properties exhibited by the wrought-iron blow-cases, it was decided to try wrought-iron factory tanks. Accordingly, all of the tanks serving as acid reservoirs at the tops and bottoms of the towers, with the exception of one wooden lead-lined tank at the top of the Glover tower for chamber acid, were made of wrought iron. Two years service has proved that the experiment was justified, as the repair account for these tanks has been practically nil, and caliper measurements of the thickness of the iron shows that the tanks have still some years to live. At No. 1 Plant, as the wooden lead-lined tanks become unserviceable, they are being replaced by iron tanks.

The storage tanks for these plants have been placed at such an elevation (with the exception of one tank) that the acid flows to them from the plant by gravity. These tanks are made of steel or wrought iron. The total storage capacity, including two tanks of 5000 tons capacity each, is 23,000 tons.

From the storage tanks the acid flows by gravity into tank cars for shipment, and with the clamping of the lid on the dome of the car, and the pasting of the diamond-shaped label on the sides and ends, the work of making a carload of acid is finished.

But while the work, with reference to any particular tank-car of acid, may be considered completed with the shipping of the car, the work of the acid-maker goes on in a cycle as endless as the flow of Glover acid to the Gay-Lussac top, down the Gay-Lussac tower to its bottom, thence as nitrous vitriol up to the Glover top, and down that tower back to the bottom, appearing again as Glover acid.

Resumption of Sulphur Production

Advices to the Chemicals Division of the War Industries Board are that mining operations have been resumed in part at the plant of the Union Sulphur Company, at Sulphur, La., where considerable damage was done by the hurricane that swept over that region on August 6. Shipments of sulphur were resumed last week from the accumulated stocks and rapid progress is being made in the work of restoring normal conditions. Considerable rebuilding will have to be done but ample material is on hand for the work.

The company carried immense reserves of sulphur above ground so that there will be no interruption in the manufacture of sulphuric acid and other necessary war materials. This reserve pile of the Union Sulphur Company measures 800 feet in length by 200 feet in width, 40 feet high, and represents about fifteen months' mining.

The plant of the Freeport Sulphur Company at Freeport, Texas, is producing its normal tonnage and has a considerable reserve above ground. While the damage to the Union Sulpher Company is unfortunate, especially at this time when sulphuric acid is being produced in such an enormous way, the reserve stock situation is such that there is no cause for worry. The Union Sulphur Company's operatives are working day and night in the rebuilding of their plant. The sulphur is melted in the ground by steam and hot water and forced to the surface where the molten sulphur solidifies on exposure to the air. The sulphur deposit underlies a bed of quicksand through which it is impossible to drive shafts and mine in the ordinary way. The development of this project is one of the interesting mechanical and chemical achievements of the past decade and, together with the Freeport Company's development, makes the United States by far the greatest producer of sulphur. There are two other known deposits which can be quickly opened in case of necessity. Work is now progressing on the development of one of these deposits.

Dyes from the Manufacturers' Standpoint

American Dye Makers Have Made Remarkable Progress in Furnishing Types of Dyes Formerly Imported—Lack of Variety of Intermediates Responsible for Failure Thus Far to Manufacture All the Dyes Desired

BY LOUIS JOSEPH MATOS, PH.D.
Chemist National Aniline & Chemical Company, Inc.

WHILE coal-tar dyes have been manufactured in the United States for many years, it has only been since the outbreak of the war that it can truly be said that dyes of strictly American origin have been produced. This point has been referred to many times by other writers, who drew attention to the fact that we do not have in this country the so-called industry of the intermediates from which American dye makers could draw their initial supplies. As a consequence, such dyes as were made in the United States were necessarily produced from intermediates that had been regularly imported from Germany.

When the war broke out, and the stocks on hand of imported and such domestic dyes as were finished were becoming rapidly depleted, the necessity arose compelling the production of intermediates from domestic raw materials and this production has gradually increased until the present time, when quite a respectable number of such commodities are now available. For some few years prior to the war aniline had been regularly manufactured at the plant of the Benzol Products Company, now the Benzol Works of the National Aniline & Chemical Co. Inc., but, it was not until after the war that secondary products, derivatives of aniline, were seriously contemplated.

Naturally in the manufacture of dyes to meet commercial conditions the manufacturers, of course, took up the production of those coloring matters that could be most readily prepared from easily manufactured intermediates, for it must be remembered that with a few given intermediates it is possible to produce quite an additional number of finished dyes. With crudes being rapidly absorbed by the explosive industry the manufacture of intermediates was put to serious disadvantage, and consequently the output of raw materials for the dye maker was extremely limited. Coupled with this was the great difficulty in securing men sufficiently trained in the chemistry and manufacturing operations. A great many men took up the manufacture of these products who did not possess a practical acquaintanceship with the intimate workings of the operations, but who by intensive application and pretenses succeeded in overcoming conditions which, under other circumstances, might have proved unsuccessful.

AN IMPOSING LIST OF INTERMEDIATES AND DYES

The industry of the intermediates as it stands today in this country is remarkable for the variety of its products, which includes an imposing list of derivatives obtained from benzol and its homologues, numbering about 30, while the list of naphthalene derivatives, including a number of the complicated naphthol and amido-disulphonic acids, is worthy of special mention. Referring to these naphthalene derivatives used as the starting point for the manufacture of a number of dyestuffs, it should be kept in mind that with very few exceptions the production of each one constitutes a distinct industry made up of a number of operations. For their proper conduct is necessary a body of men of special training, whether chemists, engineers or workmen, and whose labors are not diverted to other manufacturing operations. Their manufacture is an example of highly specialized work. Some of the chemists who undertook this work were even unfamiliar with the methods of analyzing the finished product, some were unacquainted with the appearance of the operations as one succeeded the other, all of these had to be worked out—there were many failures interspersed with a few successes. But gradually as experience was gained better and more concordant results were being secured, so that in time the quality and the output of the items were constantly increasing.

As the number of the intermediates increased, the number of dyestuffs likewise increased, but the dyer and calico printer were constantly insisting upon a wider range and better dyes and these demands consequently stimulated the dye maker, with the result that during the last forty months the number of dyes actually upon the market increased until there are now about 175 separate and distinct coloring matters of coal-tar origin available and this list is being constantly added to. It is unnecessary to record in this article the list of those dyes, but it may serve our purpose to draw attention to the fact that there are many important members of the group of acid dyes; a wide range of dyes belonging to the direct dye color group; developed colors—a small but graudally increasing number belonging to the same group; sulphur colors—the great majority of which find their application to the production of khaki and olive-drab shades for military purposes; chrome colors which produce the so-called fast shades upon wool with the aid of chromium, a notable example of which is the fast chrome blue used almost exclusively for the dyeing of cloth for sailors' uniforms; and a number of basic dyes which have always occupied a prominent position in the art of textile coloring

INDIGO AND ALIZARINE NOW PRODUCED HERE

A number of other products should not be overlooked which do not belong to the above mentioned groups. For example, synthetic indigo; there is now in the process of erection at the Marcus Hook Works, a plant of most modern construction from which will be produced at an early date very large quantities of indigo,

the process for which has been actively prosecuted for a long time and has ultimately reached the point of success where the engineers could take hold and proceed with active operation. At the Brooklyn works of the National company there is now being regularly produced alizarine, from which the dyer produces on cotton the well known Turkey Red shade, and from which may also be obtained a number of other coloring matters, broadly included in the so-called group of alizarine dyes, and by this is meant *true alizarine.*

At one time the idea of producing alizarine in this country was regarded as the most remote possibility. This idea was based upon the fact that the essential raw material for the production was anthracene, which had never been isolated from our domestic tars and without which, of course, alizarine could not be made. When it was definitely decided that alizarine would be made in this country, the separation and refining of anthracene was taken up with energy, with the result that a quality of anthracene has been produced equalling in every way that which has been produced over in England or Germany, and the resulting alizarine, according to the testimony of all dyers, compares most favorably with that which was formerly imported. The energy displayed by the National company in the production of alizarine is merely an augury as to what may be expected by dyers and colorists along the lines of other dyes which are equally difficult to produce. Dyers, as well as consumers of colored textiles, have been insitent for some coloring matters that it has not been possible for the American dye makers to produce, at least for the present. The demand has constantly been made for fast dyes and the query has even been made, Is this or that dye equal to, or as fast as, dyes formerly imported from Germany?

Our Dyes Equal in Quality to Imported

This brings forcibly to mind the fact which should not be overlooked, that no dye whether of German or of any other origin is absolutely fast. What the American dye maker tries to do is to produce dyes corresponding in type to dyes that were formerly imported. For example, let us select from the group of basic colors methylene blue. There is no question but that methylene blue possesses certain very good qualities and also certain qualities that do not condemn it for some uses, but the American dye maker has succeeded in duplicating identically methylene blue, and we are today producing in this country equal in shade, strength and working qualities the methylene blue that has been obtained from abroad prior to the war.

If we are asked, Are you making methylene blue as good as the Germans? we must emphatically answer in the affirmative. The same remarks apply to chrysoidine, the methyl violets, saffranine and Bismarck brown. If we refer to the group of sulphur colors with the same question asked us, Are you producing sulphur yellows or sulphur blues in this country equal to those formerly imported from Germany? we answer yes. But, if we are asked, Are you producing every one of such colors as those formerly imported? the answer necessarily must be No, for the reason that the industry of the intermediates has not yet kept pace with the demands. One of the most important groups of colors which interests the wool dyer particularly, is the so-called chrome colors above referred to. From this class of colors alone the dyer is in position to select quite a number that possess the good qualities of extreme fastness, and many members of this same group now being manufactured in the United States are equal in fastness to their corresponding pre-war types. If again the question is asked, Are we manufacturing in this country every member of the chrome color group? we must again answer in the negative and offering again as the reason, that the industry of the intermediates has not yet supplied to the American dye maker every necessary raw material for the production of members of this important group.

Chrome Colors for War Needs

Another reason also plays an important part, and that is military interests. With a war upon our hands and with conservation laying its hands upon wool in the interest of the army and navy, and we are likewise confronted with the fact that the fastest wool color is commandeered for military purposes and the output of the dye manufacturing plant capable of producing such color is almost solely taken over for war purposes. Coupled with this is the fact that to properly apply these chrome colors, chromium in the form of its well known chemical salts is needed, and the supply of chromium in the form of its ores is demonished so far as importations are concerned. Chromium in the form of chrome iron ore comes from widely separated localities, and while some has been mined in the United States the amount has been small in proportion to the consumption.

As used by the dyer, chromium takes the form of either bichromate of potash or corresponding soda salts. At the moment potash salts are out of the question, while soda salts are produced in limited quantities. The leather industry also requires large quantities of chromium for the chrome tanning process, producing leather for both shoes and harness, besides other military equipment. By far the small amount of chromium which comes to this country in the form of its ores finds its way directly to the steel works, where it plays its part in the war by aiding in the production of chrome steel, largely used for ordnance, projectiles, armor, etc. This disposition of the chrome ore imports is offered as a contributory reason why many chrome colors are now available to the general public

New York City.

New Method of Preparing Monomethyl Aniline and Dimethylaniline by Catalysis.—A. Mailhe and F. de Godon.—When a mixture of the vapor of aniline with a slight excess of methyl alcohol is passed over alumina heated at 400 to 430 deg. a mixture of mono and dymethyl anilines is obtained which contains only traces of aniline. If methyl alcohol is again added and the process is repeated all the monomethyl derivative is converted into dimethyl aniline. After a certain time the alumina becomes yellow and then brown and its catalytic power is slightly diminished. On calcination it again turns white and recovers its activity. In this new method of preparing the monomethyl derivative the aniline need not be very pure, but may contain water, and the methyl alcohol need not be freed from acetone. The use of autoclaves, compressed air etc. is also avoided, and labor is reduced to a minimum.—*Comptes Rendus*, Vol. CLXVI., March, 1918.

Direct and Indirect Methods of Nitrogen Fixation*

Comparison of Operations Involved—Simplicity of the Direct Method—Layout of Battery of Coke Ovens, with Electric Power House and Nitrate Plant

BY E. KILBURN SCOTT

I FEEL that the merits of the arc-flame process for making nitric acid have not been adequately and sympathetically considered, and this paper is written with the special object of stating them. I wish also to remove the misconception that the arc-flame process is dependent on water power and that it can only be installed economically on a very large scale. The matter is one of special interest to electrical engineers because the process is essentially an electrical one.

One method of producing nitric acid from air which I call the "indirect" method is first to make carbide of calcium, then treat it with nitrogen to form calcium cyanamide, from which ammonia and in turn nitric acid are obtained.

Another method is the "direct," which consists in combining nitrogen and oxygen of the air directly in the electric arc to form nitric acid.

Those interested in the indirect method have drawn comparisons between it and the direct electric arc process, with the object of showing that the indirect is the better. A tabular comparison of the operations involved in the two processes should thus prove of interest, since it is the only way in which a fair comparison can be made.

It is frequently stated that the amount of electric energy required for a given quantity of nitric acid produced by the indirect process is less than that required by the direct, and this is put forward as a strong argument in favor of the indirect method. The only way to compare two operations is to take into account all the factors which go to make up the total cost, and appraise each one at its proper value.

INDIRECT METHOD, EMPLOYING CALCIUM CYANAMIDE TO MAKE AMMONIA AND OXIDIZING THE AMMONIA TO ACID BY A CATALYST	DIRECT METHOD, EMPLOYING THE ARC FLAME FURNACE ONLY
FACTORIES	
1. To make calcium carbide.	1. To make nitric acid.
2. To make cyanamide.	
3. To make nitric acid.	
OPERATIONS	
1. Burning limestone.	1. Blowing air through electric arc flame to produce nitrous gases.
2. Grinding lime.	2. Absorption of gases in towers to produce acid.
3. Grinding coke or anthracite.	
4. Mixing lime and carbon in correct proportions.	
5. Making calcium carbide in electric furnaces.	
6. Grinding carbide to fine powder in neutral atmospheres.	
7. Making liquid air to produce nitrogen.	
8. Packing calcium carbide into retorts.	
9. Making calcium cyanamide by adding nitrogen and by use of electric resistors.	
10. Emptying cyanamide from retorts.	
11. Grinding cyanamide to a fine powder.	
12. Hydrating cyanamide to rid it of carbide.	
13. Making superheated steam.	
14. Treatment of cyanamide with steam in autoclaves to produce ammonia.	
15. Oxidation of ammonia to produce weak nitrous gases by means of a catalyst.	
16. Absorption of gases in towers to produce acid.	
RAW MATERIALS	
1. Lime.	1. Air.
2. Coke.	2. Metal electrodes.
3. Carbon electrodes in carbide furnaces.	3. Water.
4. Carbon resistors in cyanamide retorts.	
5. Pure nitrogen.	
6. Superheated steam.	
7. Air.	
8. Water.	
ELECTRIC ENERGY FOR	
1. Carbide furnaces.	1. Arc-flame furnaces.
2. Grinding carbide.	
3. Cyanamide retorts.	
4. Grinding cyanamide.	
5. Heating catalyst.	
6. Motors for power etc., including several cranes.	
SKILLED LABOR FOR	
1. Carbide furnaces.	1. Arc-flame furnace.
2. Cyanamide retorts.	2. Absorption plant.
3. Packing cyanamide.	
4. Grinding machinery.	
5. Making pure nitrogen.	
6. Making ammonia.	
7. Catalytic process.	
8. Absorption plant.	

*Abstract of a paper presented at the 34th Annual Convention of the A. I. E. E., Atlantic City, N. J., June 27, 1918.

COMPARISON OF COST OF PLANTS AND OPERATIONS

Obviously, the cost of a plant using the indirect method, will be very much greater than that using the direct, for if we assume that the cost of a carbide furnace and its accessories is about the same as that of an air nitrate furnace with its accessories, then the indirect process embraces in addition:

1. A complete plant for making cyanamide.
2. A liquid-air plant for making pure nitrogen.
3. Powerful machinery for grinding the carbide and the cyanamide.
4. Steam boilers and autoclaves for making ammonia.
5. A complete catalytic plant for oxidizing the ammonia to nitric acid.

In the indirect method it is essential to have all the materials, gases etc. absolutely pure: for example at the cyanamide works at Odda in Norway it was necessary to carry a pipe up the mountain side so as to insure a supply of pure air to the liquid-air plant.

When carbide is converted into cyanamide some of the former remains unchanged and in order to obviate danger of explosion a special treatment of the mixture is necessary to insure a total decomposition of the remaining carbide.

Platinum is usually employed and in order to "reactivate" it, it is necessary to subject it to an acid treatment and eventually to remelt, in which process it is impossible to avoid loss of this expensive metal.

By the direct method the cost of air is nil, and the cost of water is practically that of pumping. On the other hand, the materials required in the indirect method are very expensive and especially difficult to obtain at the present time. Over three-fourths of the cost of working the indirect process is represented in materials liable to price fluctuation. These are now much higher than before the war, and will remain at the higher level after the war.

In the direct method less than one-fifth of the total cost is represented in materials dependent on market rates, and the principal item of cost, namely, electric power, will, if anything, tend to come down in price.

The direct method is very simple to operate, while the indirect requires much skilled and unskilled labor, and some of the operations are dangerous to health. Therefore, the more labor demands increase, the more the indirect method will be handicapped in this respect. There are many separate links involving exact operating to make the whole run smoothly; the slightest hitch in connection with any one link necessarily holds up the whole system.

The manufacture of cyanamide has to be carried out in retorts of relatively small size involving much labor to set up etc. This is to enable the nitrogen gas to penetrate to all parts of the contained carbide. The times of the reaction and the cooling down etc. are definitely fixed and it is quite impossible to work the process with off-peak power; moreover, should there be an accident or failure of current for a time, there is every chance of ruining both the cyanamide retorts and the carbide furnaces.

All nitrate processes have a military bearing as regards preparedness, in which is involved the question of transportation. The heavy and bulky raw materials necessary for the indirect process place it at a serious disadvantage from this point of view, especially at the present time when the railways are so congested; with the direct process there is no carriage of raw materials.

As a basic load for a power house the direct arc process presents the advantage that it can be established anywhere, because, the raw materials being only air and water, considerations of transportation do not enter into the situation.

It is particularly suitable for off-peak or off-season loads, for there is no fused material to solidify and little to deteriorate in case of stoppage. Some of the furnaces can be switched on and off like an arc lamp without detriment to brickwork or structural details, or to the process of manufacture.

As there seems to be some doubt as to the possibility of running arc furnaces intermittently on a commercial scale, I would mention that about seven years ago a nitric acid factory was built at Legnano, Italy, to utilize 10,000 horsepower, especially during the night (see "Utilization of Atmospheric Nitrogen" by T. H. Norton, p. 68). Of course this plant has been considerably extended, especially since the war. I am also credibly informed that in Germany there is a very large arc-process plant working with off-peak power. At any rate there is no difficulty in doing it, whereas it is impossible to work intermittently with any other method of fixing atmospheric nitrogen.

In some ways, it is an advantage to run a plant for 8000 hours per year or less instead of the full number, because the spare time can be conveniently used for renewals and repairs. Less spare time is thus required and the plant can be operated by two shifts of men.

Small Plants Profitable

Because the plants in Norway are very large and use only hydro-electric power, a myth has spread that the arc-flame process can be worked commercially only on a very large scale, and then only with water power. As a matter of fact, it is well worth while to build plants of 10,000-kw. consumption.

Indeed, hydro-electric power may be a disadvantage because of its distance from industrial centers, for either the factory has to be placed in an out-of-the-way location, or else the power has to be carried over a long transmission line. I am of the opinion that electrochemical factories should be placed near the power supply, and the ideal position is alongside the power house, especially if off-peak power is used.

In a national emergency it is surely better to bring into immediate use all the surplus equipment that already exists than to start building new power houses, whether hydraulic or steam, and inasmuch as the direct-arc-flame process is suitable for working with off-peak power, I would suggest that a number of nitrate plants be forthwith erected at existing power houses.

By erecting ten or more nitrate plants of 10,000 kw. each at power houses near places where nitrates are required, there would be considerable saving in transportation; early deliveries of nitrate could be made. Furthermore, there would be less risk of temporary interruption of supplies in case of accident or sabotage.

There are power houses which could easily spare more than 10,000 kw. for over 20 hours a day and over the week end. Moreover, there are power houses fully equipped with steam plants which are now standing idle. In the present crisis they might just as well be brought into use even if the cost of generation is high.

In some power houses the load factor might be doubled; this would have the immediate effect of reducing costs. But there has been far too much shilly-

shallying consideration given to questions of cost. With U-boats on the high seas trying to stop supplies of Chile nitrate, the railways congested with traffic and electrical engineering works making munitions, what is the use of discussing power costs?

Coke-Oven and Nitrate Plants

At the present time ammonium nitrate is required in very large quantities for burster charges for shells, torpedoes, mines, grenades etc. This is made from two components, viz., nitric acid and ammonia, both of which are difficult to transport, the first because it is a corrosive acid and the second because to every ton of aqua ammonia there are about 2½ tons of water. An industrial process capable of furnishing electric energy as well as a supply of ammonia would be ideal, and it so happens that this is the case with a regenerative coke-oven plant. Half the total gas made is available and this can be easily turned into electric energy while at the same time the nitrogen contained in the coal provides about the right amount of ammonia necessary to combine with the nitric acid made from the electric energy by the arc-flame process.

In order to show how ideal such a system is for making ammonium nitrate, I have prepared the diagram, Fig. 1.

The scheme provides for a combination of a battery of coke ovens with an ammonia-recovery plant together with an electric power house in order to utilize

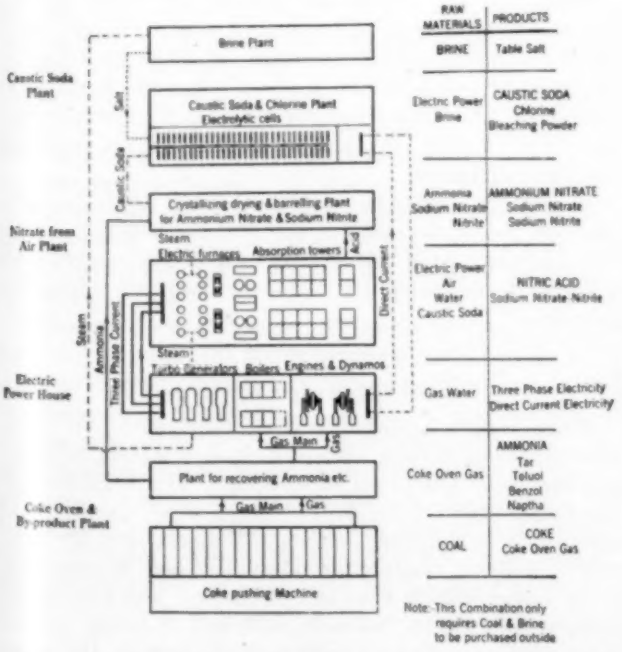


FIG. 1. SYSTEM FOR MAKING AMMONIUM NITRATE

the surplus gas. Alongside of the power house, there is an electrochemical plant for the manufacture of nitric acid from air by utilizing the three-phase high-tension current. A nitrate house is provided for the purpose of combining the ammonia from the coke ovens with the nitric acid from the electrochemical plant.

It happens that the by-product of the acid factory is sodium nitrate-nitrite, which is made by combining the gases remaining from the acid towers with caustic soda or soda ash. Electrolytic cells may be laid down as shown in the diagram for the purpose of making caustic alkali from brine.

It will thus be seen that the complete project requires only two raw materials, viz., coal and brine, and on the other hand, the products which can be made are coke and ammonium nitrate, together with toluol, benzol, naphtha, tar and sodium nitrate-nitrate.

If electrolytic cells are used, there are also the products chlorine and bleaching powder. The chlorine can be combined with the benzol to form chlor-benzol which is an important intermediate in the manufacture of dyestuffs as well as in the manufacture of picric acid.

From the point of view of efficient management and elimination of transportation charges, the combination is unique, for the ammonia has to be piped only a few yards to the nitrate house and there is no carriage of acid.

As a cheap supply of coal is indispensable for the project, it would be well to locate the plants at industrial centers where this raw material is readily available and where in all probability transportation charges would be low.

Yield of a 100-Oven By-Product Plant

In order to show what can be done with a coke-oven plant the following particulars will be of interest. I take a Koppers type of oven as being the best known.

Quality of Coal	Tons per charge	Hours coking time
Low-volatile coal	13½	18
Mixture containing 80 per cent high-volatile, 20 per cent low-volatile coal	12½	16½
High-volatile coal	11½	15

A battery of ovens varies in size but we may as well take a round number of 100, for which the average yields are as follows:

Number of ovens	100
Tons of coal per oven	12½
Hours coking time	16
Total yield of coke, per cent	72
Yield small coal and breeze, per cent	5
Net yield good coke, per cent	67
Ammonium sulphate per ton of coal, lb	25
Reckoned as ammonia per ton of coal, lb	6½
Tar per ton of coal, gal	9
Light oil per ton of coal, gal	3
Total gas per ton of coal, cu.ft	11,000
British thermal units, per cu.ft	550
Surplus gas, per cent	55
Surplus gas per ton of coal, cu.ft	6,000

Such a battery of ovens, each of which distils 12½ tons of coal in 16 hours, will deal with

$$\frac{100 \times 12.5 \times 24}{16} = 1900 \text{ tons per day.}$$

Assuming 6000 cu.ft. of surplus gas per ton of coal and 550 B.t.u. per cu.ft., the total heat value per hour will be

$$\frac{1900 \times 6000 \times 550}{24} = 260{,}000{,}000 \text{ B.t.u.}$$

If employed in gas engines using 13,000 B.t.u. per hp.-hr., the power will be

$$\frac{260{,}000{,}000}{13{,}000} = 20{,}000 \text{ hp., or about } 14{,}000 \text{ kw.}$$

If steam boilers and turbines are used instead of gas engines, the power will be less; so to be on the

safe side, we will take the round figure of 10,000 kw.

We will also assume that electric furnaces utilizing 10,000 kw. for a whole year can produce 6300 tons of 100 per cent nitric acid capable of furnishing theoretically 8000 tons of ammonium nitrate, as indicated:

	NH_3	+ HNO_3	= NH_4NO_3
Molecular weights	17	63	80
In short tons	1700	6300	8000

Allowing 25 lb. of sulphate of ammonia or 6½ lb. of ammonia per ton of coal, a total consumption of 1900 tons of coal per day should give

$$\frac{1900 \times 365 \times 6.5}{20{,}000} = 2250 \text{ tons per annum.}$$

It will thus be seen that there is plenty of ammonia to combine with the acid made by the surplus gas, even if a higher yield of acid is allowed per kw.-yr. and more power is generated.

I purposely leave out of consideration questions as to types of nitrogen fixation furnaces and of yields obtained. I may say, however, that it is not right to assume that yields are limited to those usually obtained from certain well-known furnaces which must of necessity work with single-phase current.

The amount of ammonium nitrate will be less than the theoretical figure because the efficiency of the reaction is not 100 per cent; moreover, it is usual to convert a certain amount of the gas into sodium nitrate-nitrite. A safe figure would be 6000 tons and at this rate it can be shown that with electric energy at 5 mils per kw.-hr. and ammonia at 13c. a pound, the ammonium nitrate can be made at less than half the price the government is now paying.

Magnitude of the Nitrogen Industry

In order to show how large a business the nitrogen industry has become, the following figures (compiled by Dr. Paul J. Fox) give the nitrogen balance sheet for the United States for 1917.

	Tons of 2,000 lb.	Tons of Nitrogen
IMPORTED SUPPLIES		
Chile saltpetre, 95 per cent $NaNO_3$	1,742,540	272 [illegible]
Ordinary saltpetre, potassium nitrate	4,609	645
Ordinary saltpetre and gunpowder containing 75 per cent KNO_3	1,500	210
Ammonium sulphate	8,135	1,725
Ammonium chloride	1,073	
DOMESTIC SUPPLIES		
Coke oven ammonia—NH_3	113,760	93,625
Gas works ammonia—NH_3	12,500	10,288
Calcium cyanamide at 20 per cent nitrogen	12,800	10,534
NITROGEN EXPORTED		
Nitric acid, 15 per cent nitrogen	486	73
Picric acid, 18 per cent nitrogen	26,610	4,790
Dynamite, 12 per cent nitrogen	8,962	1,255
Gunpowder and smokeless powder, 13 per cent nitrogen	223,270	29,025
Ordinary saltpetre	875	123

In addition to the above, there is also about 8800 tons represented as nitrogen in the following items which are the figures for 1917.

	Value
Loaded cartridges	$42,000,000
Fuses	34,000,000
Shells and projectiles	74,000,000
All other	202,000,000
Total	$253,000,000

It will be noticed that ammonium nitrate is not included in these figures, but I assume it would be about 50,000 tons for 1917.

In Great Britain the consumption of ammonium nitrate is now probably 400,000 tons a year, and the production here will have to be at least as much. To make this, the theoretical proportion of ammonia required is about 85,000 tons and of nitric acid about 315,000 tons.

It will thus be seen that the coke-oven plants in the country could supply all the ammonium nitrate required if they were put on the job.

General Considerations

Until recently most coke-oven ammonia was converted into sulphate, but owing to the war demand for nitrate, more and more of it is being made into aqua ammonia of about 29 per cent strength. In some cases this is being transported many hundreds of miles prior to conversion into ammonium nitrate and since each ton of ammonia necessitates the transportation of about 2½ tons of water, the bearing of this on the present railway congestion is at once apparent. Tank cars have to be used and they must return empty, so the freight on the actual ammonia carried is extremely high.

There are many coke ovens of the wasteful bee-hive type in operation, which do not recover by-products. The replacement of these by modern coke-ovens would be a great immediate economic gain and meet the war conditions better than the building of large dams for water power.

In the present emergency coke ovens are of great value because they give coke for making steel, gas for power purposes, ammonia for nitrate manufacture, and toluol and benzol for explosives.

After the war ammonium nitrate will be in demand for fertilizer as well as for safety explosives and other purposes. The high percentage of nitrogen which it contains, 35 per cent, and the ease with which it can be converted into other compounds makes it especially useful for conveying nitrogen in the fixed form over considerable distances.

It is more profitable to make nitrate than sulphate, because pound for pound, the nitrate contains nearly twice as much fixed nitrogen and the nitrogen commands a higher price per unit when in the form of ammonium nitrate.

New York City.

Jellies Formed by Dyes

When certain substantive dyes, Benzopurpurin B and Chrysophenin B, are dissolved in hot water, solutions are obtained which jell on cooling. The formation of the jellies seems to be dependent on small quantities of electrolyte in the dyes, for when the solutions are purified by dialysis, coagulation takes place and the coagulum is no longer soluble even in hot water. The addition of sodium chloride is, however, sufficient to bring the dye into solution. The experiments show that the small quantities of electrolytes (sulphates and chlorides) present in the commercial substantive dyes are very important in technical application, but as yet no satisfactory explanation of the phenomenon can be given.—*Kolloid Zeits.*

The volume of natural gas from which the output of natural-gas gasoline in 1917 was recovered is estimated to have amounted to about 429,000,000,000 cu. ft., and the average recovery of gasoline per thousand cubic feet was about one-half gallon.

The Distillation of Coal in a Vacuum*

Nature of Products Obtained and Their Relation to Ordinary Tar and Petroleum in Chemical Constitution

By AMÉ PICTET
Professor of Chemistry, University of Geneva

WHEN we consider the part that coal plays in the modern world, we would be inclined to the belief that this precious material had long been the subject of thorough and definitely scientific study, and that its chemical nature particularly was no longer a secret. As a matter of fact, this is not true. The chemistry of coal still presents considerable blanks. If we know accurately its elementary composition and the calorific power of all the varieties, if we know exactly all the products which it yields upon heating, we are still ignorant of what it is in its natural state and of what organic bodies it is composed.

This knowledge would be very important to science and industry, for it would render possible the solution of a number of important questions which are still unsolved and which have a bearing upon, among others, the genesis of fossil coals and their relation to petroleum and bitumens, upon the slow transformations to which they are subjected in the earth and to those more rapid transformations which they undergo during cokefaction, etc.

The ignorance in which we are left in this domain, however, must not be imputed to the indifference of the chemists; it is attributable only to the insufficiency of the means at their disposal. To separate the constituents of an intimate mixture, they have in effect but two processes: Fractional distillation and the action of solvents. Now, neither the one nor the other is practicable in this particular case, for coal is not volatile without decomposition and does not dissolve easily in any known liquid.

In spite of these inherent difficulties of the subject, I have thought that the latter deserved to be undertaken and that it would be possible to overcome some one of the obstacles by changing suitably the operating methods used up to the present time. Several years ago with the assistance of several co-workers, MM. L. Ramseyer, M. Bouvier, O. Kaiser and A. Labouchère, I entered upon some original research upon the chemical nature of coal. These studies are far from being finished; their first results have led however to some conclusions which may perhaps be interesting.[1]

Method of Research

We have been guided in our first experiences by the following considerations. When coal is distilled at atmospheric pressure as is practiced in gas works and in coke-plants, it is necessary, in order to extract all the volatile properties, to heat it to a very high temperature (800 or 1000 deg.). It is evident, that under these conditions, the products that are collected, and which form the illuminating gas, the ammoniacal water and the tar, must be considered as pre-existent in the coal, and not as resulting from its immediate decomposition. They have undergone, due to the high temperature to which they have been brought, various transformations, the nature of which has escaped us. We cannot deduce from their chemical constitution any index as to the more complex compounds from which they have been derived.

There is only one way to avoid these transformations and that is to conduct the distillation at a much lower temperature and at a very reduced pressure. Acting upon this principle, we have subjected to vacuum distillation several kilos of a fat coal coming from Montrambert (Loire). The simple apparatus which we used has been described elsewhere, and I shall not repeat it. We have found that at a pressure of 15 mm. of mercury, distillation begins at about 100 deg. and that it is entirely terminated at 450 deg., that is to say, a little above the point where it would begin if operated under ordinary pressure.

Nature of Products Obtained

The products obtained are of an entirely different nature and are as follows:

1. *Gases.* The apparatus which we adopted did not permit us either to measure them or determine their composition; we have only been able to observe that their odor is much different from that of illuminating gas and resembles that of butadienes and isoprenes.
2. *Water.* About 1.6 per cent of the weight of the coal presented an acid reaction and did not contain ammonia.
3. *Tar.* About 4 per cent; very different from ordinary tar in appearance and all its properties.
4. The *coke,* lighter, more friable and more easily combustible than ordinary coke, resembled rather that which remains after the distillation of petroleum.

Of these products, the tar seemed to be the most interesting, and we studied it first. To procure a sufficient quantity, we applied to the Society of Chemical Industry at Bâle which was so kind as to distill in a vacuum for our purpose one and one-half tons of the same Montrambert coal, which yielded us about 60 kilos of tar.

The Vacuum Tar

The vacuum tar, as we have called it, is a translucent liquid, light brown and has a light green fluorescence; it is more fluid than ordinary tar and has an odor very different, resembling that of petrol. Immediately after its preparation it floats upon the water which it has entrained but it soon thickens, sinks and increases in

*Translation from *Revue Generale des Science.* Oct., 1916, Vol. 27, pp. 578-584.

[1]For experimental details, see *Comptes Rendus,* vol. CLVII, pp. 779 and 1436; vol. CLX, p. 629, as well as the thesis of M. Ramseyer (Geneva, 1911) and M. Bouvier (Geneva, 1915).

density. At the end of three days, the density is exactly equal to 1; it rises again eventually. This phenomenon takes place when the air is excluded as well as in the presence of air; it is not, therefore, due to oxidation.

In connection with this fact, it was observed that the tar agitated soon after its preparation with an excess of solution of caustic soda does not give up anything. Therefore it does not contain phenols, but the latter appear after a certain time and their quantity increases quite rapidly.

Treated with dilute hydrochloric acid, the vacuum tar yields a small quantity (about 0.5 per cent) of compounds of a basic nature. Their study has been undertaken by M. Labouchère. Some of these substances are secondary bases, and others tertiary. These belong chiefly to the hydropyridic and hydroquinolic series. The only two of which have been isolated thus far in their pure state have the formulae, $C_{10}H_{11}N$ and $C_{12}H_{15}N$ and very probably constitute dihydromethlquinoline and dihydrotrimethylquinoline.

After this double treatment by soda and by hydrochloric acid, the vacuum tar still contains some oxygen compounds (about 2 per cent). These can be obtained by boiling the liquid with sodium. The compounds have some of the properties of the alcohols. MM. Bouvier and Kaiser have been able to isolate by fractional distillation four well-defined representatives of this class of bodies. They answer to the formulas, $C_7H_{11}O$, $C_8H_{13}O$, $C_9H_{15}O$ and $C_{10}H_{17}O$.

The first of these alcohols is saturated and constitutes the p-methyl-cyclohexanol (hexahydro-paracresol). The other three which form a homologous series belong to a group of unsaturated alcohols. Their structure has not yet been established; we have only ascertained that they are but slightly stable. Completely insoluble at first in alkalies, they become part soluble after a short time; the solution is disturbed when a current of carbonic gas is passed through it, and one can distinguish bodies having the odor and all the properties of phenols. This transformation which is probably isomerisation, furnishes the key to the phenomenon which has been a baffling question.

Once rid of bases and alcohols, the vacuum tar is nothing more than a mixture of hydrocarbons, of which some are saturated and others are not. To separate them we have made use of the Edelneau process, which has been employed for several years in the refining of petroleum. It consists in agitating the mixture, suitably refrigerated, with twice its volume of liquid sulphurous anhydride. Only the unsaturated hydrocarbons pass into solution and can be recovered simply by volatilization from the solvent. The saturated hydrocarbons serve to float the sulphurous solution and are separated mechanically. This process, applied to vacuum tar, has given us excellent results and has permitted us to separate our product very nicely into two parts, the one, unsaturated, the other saturated, the first having a volume about double that of the second.

We afterward subjected these two parts to a series of fractional distillations two degrees apart. The two parts behaved almost the same. The distillation began at 120 deg. and ended at 350 deg. There remains a small quantity of a black material of the consistency of asphalt.

All these fractions up to 300 deg. remain liquid after cooling. None of them deposits crystals. This indicates that no noticeable quantities of naphthaline, anthracene or other aromatic solid hydrocarbons are found. We have oxidized many of them by means of potassium permanganate and have found but few acids of the fatty series (acetic, proprionic, butyric, oxalic). It seems very certain that vacuum tar contains no phenols nor any more hydrocarbons of the aromatic series.

We have studied the lower fractions especially. They are liquids, insoluble in water and volatile with it. They have the odor of petroleum and are inactive to polarized light. M. Bouvier has determined their composition, their molecular weight, their density and their refraction indices and has succeeded in determining the constitution of some by converting them into known bromine and nitrogen derivatives. These observations establish the presence of the following compounds in vacuum tar:

Saturated Hydrocarbons. These all have the same centesimal composition corresponding to the general formula C_nH_{2n}; they are therefore cyclanes:

C_9H_{18}, boiling point 135-137 deg. (hexahydro-mesitylene)
$C_{10}H_{20}$, boiling point 172-174 deg. (hexahydro-durene)
$C_{11}H_{22}$, boiling point 189-191 deg.
$C_{12}H_{24}$, boiling point 211-213 deg.
$C_{13}H_{26}$, boiling point 227-229 deg.
$C_{20}H_{40}$, boiling point 218 deg. at a pressure of 0.5 mm.

The last hydrocarbon is solid at ordinary temperature and is deposited with the last fraction (above 300 deg.). It crystallizes in acetone in pearly scales, melting at 62-63 deg. We have found it identical with melene which Brodie separated in 1849 from the products of dry distillation of beeswax.

Unsaturated Hydrocarbons. These have been isolated to date in a pure state, as dihydro-trimethylbenzene, C_9H_{14}, boiling at 163-164 deg., and a hexahydrofluorene, $C_{13}H_{18}$, boiling at 240-250 deg., and it is very probable besides that the lower fractions contain dihydrogenous derivatives of metaxylene, of mesitylene and of pseudocumeme for they yielded these hydrocarbons by careful oxidation.

Relation of Vacuum Tar to Ordinary Tar

It is recognized that all the constituents of vacuum tar, whose nature we have been able to determine, belong to the hydro-aromatic series. The aromatic compounds of ordinary tar are replaced in our product by their hydrides. The homologs of benzene make place for those of cyclohexane and cyclohexadienes, the phenols for the alcohol, the quinoleique bases to their dihydrogenic derivatives.

It is seen that these hydrides when they are carried to a high temperature, have a tendency to lose a part of their hydrogen, to be converted into more stable aromatic compounds. It was then assumed that a phenomenon of this kind takes place in the gas retorts and that the hydrides of vacuum tar first form here and undergo with a higher temperature an immediate pyrogenous dehydrogenation. Vacuum tar would represent then an intermediate stage in ordinary tar formation.

This supposition was easy to verify by test. We took 200 g. of vacuum tar in its raw state, that is to say, not yet separated from its alcohols and bases, and caused it to run drop by drop into a red-hot iron tube,

first filled with pieces of coke (in order to realize as much as possible the conditions of the gas retort). At the other end of the tube we collected:

1—Gases (20-30 liters). These had the odor of illuminating gas and burned with a brilliant flame; they are composed of hydrogen and methane, accompanied by a little ethylene, but no acetylene.

2—Water strongly charged with ammonia.

3—A tar having the appearance and the odor of ordinary tar and from which we were able to separate benzene, toluene, xylenes, phenols, a small amount of naphthaline, anthracene and pyridic bases.

These are the principal constituents of ordinary tar. Their absence having been duly proved in the vacuum tar, the hypothesis which we have announced seems to us to be justified; the hydrogen and the methane of illuminating gas, the ammoniacal water of illuminating gas, the phenols and the aromatic hydrocarbons of tar, are not at all the immediate products of the dry distillation of coal. They are only formed at high temperatures by the decomposition of other more complex volatile compounds, and in particular the more hydrogenated ones which are formed at a lower temperature.

Contradiction of Berthelot's Theory of Coal Tar

This interpretation is in contradiction with the theory of Berthelot on the formation of coal tar. It is known that the latter theory consists in admitting that upon distillation the coal is decomposed entirely into very simple products—carbon, hydrogen, water vapor, ammonia, methane, ethylene, acetylene. On contact with the cold walls of the retorts, the last of these gases, notably acetylene, undergoes a series of polymerizations and condensations which give rise to diverse aromatic hydrocarbons of tar. This is also a product of synthesis.

Berthelot has based this theory, not only on a direct study of the decomposition of coal, but on experiments of pyrogenation to which he has submitted the acetylene itself. These experiments which have been repeated recently on larger scale, but with an identical result, by M. R. Meyer at Brunswick, have established the fact that the acetylene is polymerized to a dark red and yields benzene, and that the two hydrocarbons then unite in turn to produce naphthaline and that finally it is possible to obtain by means of pyrogenated condensation of the same kind almost all of the hydrogen carbides which are found in tar.

However interesting these syntheses may be, should it necessarily be deduced that they are brought about in the gas retorts? One can only assume this, it seems to me, when it has been proved that the coal upon heating liberates great quantities of acetylene. Now this proof has not, to my belief, yet been established.

This argument would not constitute however serious objection to the theory of Berthelot even if it had been corroborated by some recent experimental facts. Perhaps it will be accepted that our study of vacuum tar supports some of these facts, and that the new explanation which I propose merits substitution in a large measure waht has held up to the present time.

I say in a large measure, for my interpretation, it seems to me, ought to be applied to the major part of the reactions which take place during the distillation of coal, since this yields almost the same quantity (4 per cent) of vacuum tar as ordinary tar. However, I realize that this does not yet account for all of these reactions. Moreover, we have not been able to discover in our tar any of the hydrogen derivatives of naphthaline. This hydrocarbon, if it exists in ordinary tar, must then have a different origin than the others, and it is highly probable that it originated through a synthetic process similar to that which Berthelot admitted. The following observation seems justified in clearing up this point. We have found that a very considerable quantity of naphthaline was formed when the vapors of cyclohexane are conducted over coke heated to redness (the vapors of benzene under these conditions yield only biphenyls). Since the cyclohexane reappears in the series of the saturated hydrocarbons which we have derived from vacuum tar, there can be seen in this reaction the source from which the naphthaline of ordinary tar proceeds.

Relations of Vacuum Tar to Petroleum

Allusion has been made in the preceding pages to analogies that vacuum tar present to petroleum. These analogies have been noticeable not only in the physical properties of the two liquids, but also in their chemical composition. After having isolated the series of cyclanes mentioned above, M. Bouvier and I compared their properties with those naphthenes which form the essential part of certain petroleums. This comparison has shown us that the naphthenes of Baku petroleum are not identical with the cyclanes of vacuum tar. For an identical formula, they have a higher density and a higher boiling point. On the other hand, we have found a remarkable similarity between the properties of our hydrocarbons and those which M. Mabery[1] attributes to the naphthenes which he has derived from certain petroleums from North America (Canada and California). Table I shows the results of his investigation.

TABLE I—RELATION OF VACUUM TAR TO PETROLEUM

Vacuum Tar				American Petroleums				
Fraction Deg.	Formula	Density	Index of Refraction	Fraction, Deg.	Formula	Density	Index of Refraction	Source
135–137	C_9H_{18}	0.7590	1.4212	135	C_9H_{18}	0.7591	.....	Cal.
172–174	$C_{10}H_{20}$	0.7765	1.4196	173–174	$C_{10}H_{20}$	0.7770	1.4149	Can.
189–191	$C_{11}H_{22}$	0.7838	1.4234	189–191	$C_{11}H_{22}$	0.7832	1.4231	Can.
211–213	$C_{12}H_{24}$	0.7862	1.4293	212–214	$C_{12}H_{24}$	0.7857	1.4289	Can.
229–237	$C_{13}H_{26}$	0.7953	1.4379	228–230	$C_{13}H_{26}$	0.7979	1.444	Can.

It cannot be doubted in considering the results set forth in this table but that there has been found here unmistakably these identical hydrocarbons. I might add that we have separated from the last fractions of distillation of Galician petroleum a solid hydrocarbon of the formula $C_{30}H_{60}$ which was shown to be identical in all its properties with the melene of vacuum tar.

We determined also the unexpected fact that we could separate from coal, by simple distillation, a series of compounds which constitute an integral part of certain petroleums. It is the first time, we believe, that there has been found a relation of a chemical nature between these two natural combustibles.

Relation of Vacuum Tar to Coal

One point remains to be cleared up: Do the various compounds which form vacuum tar exist as such in the coal, or are they produced during the process of distillation?

[1] *Journal of the American Chemical Society*, Vol. XIX, p. 470; Vol. XXV, pp. 267 and 276; Vol. XXXIII, p. 264.

To be able to answer this question, it is necessary to revert to the methods of exhausting coal by appropriate liquids. Considerable work has already been done on this subject. Since the time of Comines de Mareilly (1862), it has been pursued almost without interruption down to the present time. Investigators have been especially concerned, however, with establishing the relative power of extraction of various solvents, and their authors have not succeeded up to the present in separating from the extracts any definite bodies of which they have been able to determine the structure or even the composition.

I have undertaken these experiments with MM. Ramseyer and Kaiser. As a solvent, we chose benzol. We then worked with small quantities of this same Montrambert coal, which was used in our experiments with vacuum distillation. Later, thanks to the valuable assistance rendered us by the house of F. Hoffman, La Roche and Company at Bâle, the same operation was repeated on a larger scale (with 5½ tons of coal); at that time, however, a Sarre coal (Maybach) was used in the work.

Boiling Benzol as a Solvent

In the two cases the coal, crushed in pieces the size of a pea, was treated with boiling benzol for four days in the apparatus of the Soxhlet system. The solution was immediately reduced to a strong concentrate and to it was added five times its volume of petroleum ether (boiling point 35-90 deg.). It precipitated a clear brown amorphous powder, which we have not yet studied. The solvent was then driven off by heating to 100 deg. There remained a pale brown liquid, somewhat fluid, possessing an odor of petroleum and a density exactly equivalent to 1.000 at 20 deg.

The five tons treated at Bâle yielded 2.7 kilograms of solid substance and 10.6 kilograms of liquid extractive. The yield was then 0.25 per cent.

The liquid extract obtained from the Montrambert coal was especially studied from the point of view of the less volatile substances which it might contain. Without entering into more of the details of this part of our study, I will simply say that we are able to separate the same hexahydrofluorene $C_{13}H_{18}$ and the same melene $C_{30}H_{60}$ as was found in the vacuum tar.

As to the liquid extract of the Sarre coal a large quantity of which we had on hand, we decided, on the other hand, to study its more volatile products. This extract possessed not only the odor and density of vacuum tar but also a very similar composition. It was formed, like it and in similar proportions, of saturated and unsaturated hydrocarbons and about 2 per cent alcohols and bases. In separating these bodies we proceeded in exactly the same manner as we had formerly, that is to say, by successive treatments with hydrochloric acid, sodium, liquid sulphurous anhydrides, then fractional distillation.

The definite hydrocarbons which we have isolated up to the present are given in Table II. M. Kaiser was able to determine the structure of the first three by converting them into known nitrogen and bromine compounds, and was able to establish the identity between the last three compounds and the cyclanes of the same formulas separated from the vacuum tar and the petroleums of Canada.

It is evident then that there can be extracted from the coal by means of boiling benzol (80 deg.) a series of hydrocarbons which are indentical or very analogous to those yielded by vacuum distillation. This last operation then only separates them by simple volatilization, and we are safe in concluding that a part at least of the components of vacuum tar exist already formed in the coal.

They occur then as a solid hydrocarbon substance impregnated with a liquid chemically akin to petroleum. That this liquid has been formed through a slow decomposition of the solid mass, is the probable explanation, when we consider the formation of fire-damp which has been proved in all coal deposits, as well as the existence of the natural gas sources which are sometimes located in the proximity of these deposits. Fire-damp and natural gas are mixtures of hydrocarbons.

TABLE II.

Unsaturated Hydrocarbons			
C_7H_{10}	boiling point	108–110 deg.	Dihydro-toluene
C_8H_{12}	boiling point	135–137 deg.	Dihydro-metaxylene
C_9H_{14}	boiling point	166–168 deg.	Dihydro-mesitylene
$C_{10}H_{16}$	boiling point	180–182 deg.	Dihydro-prelinitol (?)
$C_{11}H_{18}$	boiling point	200–202 deg.	
$C_{14}H_{16}$	boiling point	236–238 deg.	
$C_{13}H_{12}$	boiling point	251–254 deg.	Dihydro-fluorene (?)
Saturated Hydrocarbons			
C_nH_{16}(?)	boiling point	118–122 deg.	The analysis gave us figures a little too high as to hydrogen content, thus indicating the presence in slight amounts of hydrocarbons of C_nH_{2n+2} formula.
C_9H_{18}(?)	boiling point	135–138 deg.	
$C_{10}H_{20}$(?)	boiling point	172–174 deg.	
$C_{11}H_{22}$	boiling point	190–192 deg.	Identical in every respect with the hydrocarbons of the same formulae obtained from vacuum tar and petroleum from Canada.
$C_{12}H_{24}$	boiling point	215-216 deg.	
$C_{13}H_{26}$	boiling point	227–229 deg.	

It is possible to conceive that near these gaseous products the spontaneous decomposition of coal could form from them others of the same chemical nature but which at the surrounding temperature are liquid and slightly volatile. They would remain necessarily embedded in the solid mass, from which vacuum distillation or even a simple washing with benzol could separate them.

We could go even farther and couple the origin of actual petroleum deposits to a decomposition of this kind which will come in due time. But this idea meets with numerous objections of a geologic nature. Its discussion in the present state of our knowledge would be premature. I prefer then not to touch upon this point here, and to limit myself to this short resumé of the experimental research which my co-workers and I have done on the coal itself. I believe it to be my duty to refrain for the time being from considering the industrial applications which our methods may receive. They will appear better when our research has become farther advanced.

The Natural Indigo Industry.—With the cutting-off of the supplies of synthetic indigo from Germany since the outbreak of war, the cultivation of natural indigo has increased considerably. While the total area under this crop in 1914-15 was only 148,400 acres, in 1915-16 it rose to 353,100, and in 1916-17 to 756,400 acres. The greatest increase, both relative and absolute, occurred in the Madras Presidency and in the United Provinces, where the industry is mainly in the hands of small holders, and the dye manufactured is of an inferior quality.